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Including the Proceedings of the
AMERICAN ASSOCIATION OF TEXTILE CHEMISTS AND COLORISTS

VOLUME XIV JUNE 29, 1925 NUMBER 11

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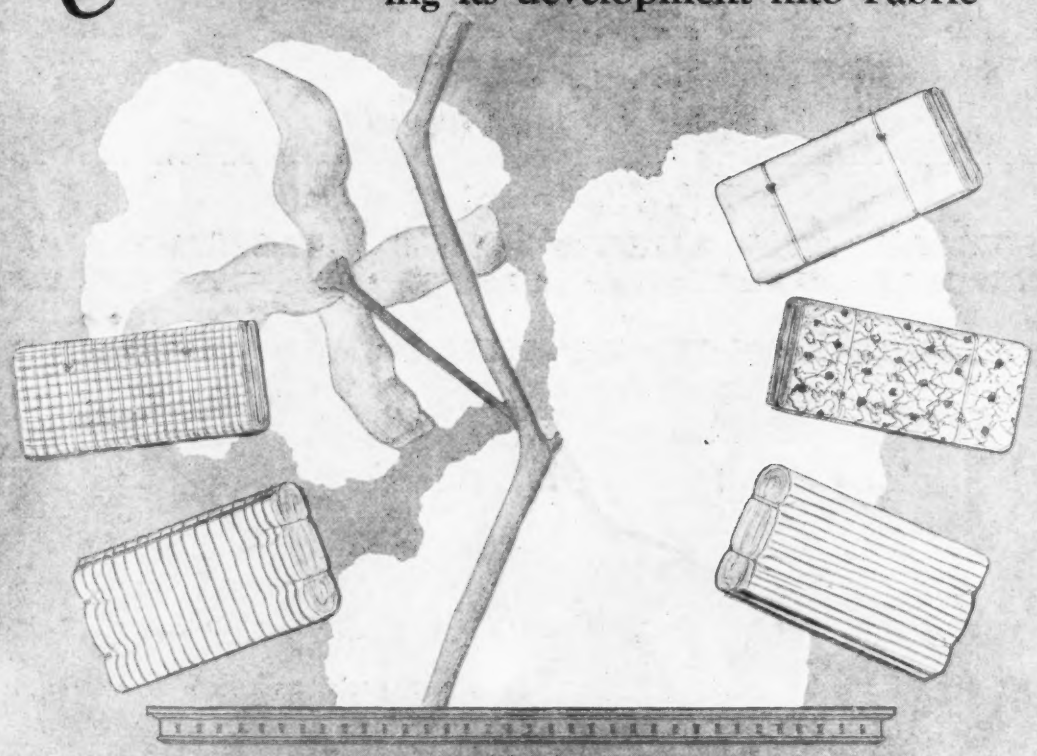

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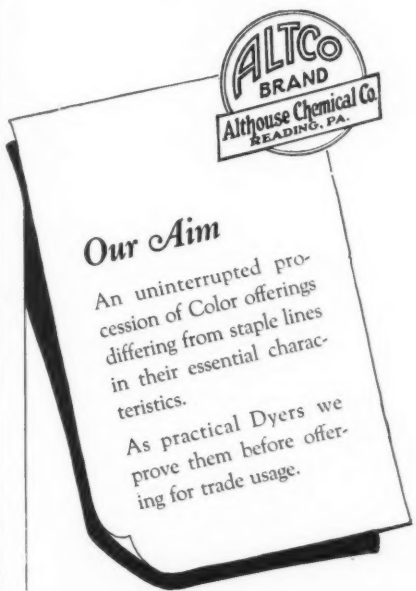
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VOLUME XIV NEW YORK, JUNE 29, 1925 NUMBER 11

Analysis of Dye Mixtures by Means of Titanous Chloride

One Hundred and Fifth Contribution from the Color Laboratory of the Bureau of Chemistry, Washington, D. C.

By WALTER C. HOLMES[1]

ALTHOUGH titanous chloride has found extensive application in the evaluation of individual dyes, it seems never to have been used in determining the percentage of the components of dye mixtures. The investigation here reported is a preliminary survey of the possibilities in this field.

The titration of dyes by means of titanous chloride is usually conducted in the presence of tartrates, or other catalytic agents[2], which greatly facilitate the reduction of most dyes. Under these conditions dye mixtures ordinarily undergo complete reduction readily and with but slight evidence of selective action. Under less generally favorable conditions, however, one or the other of two types of selective reduction, which may be utilized as an important aid in the analysis of simple dye mixtures, are met. With suitable provisions both components of a dye mixture may be reduced, but the reduction of one component may take precedence over that of the other, or the conditions may allow the reduction of only one of the components.

Selective reduction of the first type was noted by Knecht and Hibbert[3] with mixtures of azo dyes. It is not confined to azo dyes, but is found in all classes of dyes which can be reduced by means of titanous chloride. With dyes in general the character of the reduction and the nature of the reduction products do not render the phenomenon adaptable to the requirements of quantitative analysis. With quinonimide derivatives, however, it provides a satisfactory means of analysis..

Quinonimide dyes are readily reduced in strongly acid solutions by titanous chloride, even at room temperatures. A number of azine, oxazine and thiazine dyes are arranged in the following list in the relative order of the facility of their reduction in 33 per cent acetic acid.

1. Thionine (C. I. 920)[4].
   Methylene Violet (dimethyl thionolin).
2. Printing Violet R (C. I. 888, note).
3. Methylene Blue (C. I. 922).
   Cresyl Fast Violet (NAC).
   Brilliant Cresyl Blue (C. I. 877).
   Thiocarmine (C. I. 928).
4. Rhoduline Violet (C. I. 844).
   Methylene Violet RRA (C. I. 842).
   Ethyl Blue BF (C. I. 854).
   Naphthazine Blue (C. I. 855).
5. Safranine O (C. I. 841).

(The order of dyes within the various groups is without significance. It is probable that the differentiation could be made more precise by more refined methods, which were not available in the present investigation.)

Each dye listed is easily reduced not only by titanous chloride but also by the leuco compounds of the dyes in the groups below. Methylene Blue, for example, is reduced by leuco Safranine, and Thionine by leuco Methylene Blue. Conversely, the leuco compound of each dye listed is readily oxidized by the dyes in the groups above. In the titration of a mixture of Methylene Blue and Safranine, accordingly, the reduction of the Methylene Blue proceeds to completion before any possible reduction of the Safranine; likewise the reduction of Thionine precedes that of Methylene Blue with mixtures of the latter dyes. After reducing one component it is necessary only to continue the addition of titanous chloride in order to reduce the second. The record of two such analyses

[1] Associate Chemist, Color Laboratory, Bureau of Chemistry. This paper was presented before the Dye Section of the American Chemical Society at its meeting in Baltimore, Md., April 6 to 11, 1925.

[2] Piccard, Ber. (1909), **42**, 4341.

[3] "New Reduction Methods in Volumetric Analysis," London, 1918 edition, pages 7 and 8.

[4] These are list numbers of the new Colour Index of the Society of Dyers and Colourists.

(Series 1 and 2) is given in the table of analytical data.

The analytical utility of selective reduction of the second type is more diverse and comprehensive. Although the individual dyes of various classes show a wide diversity in their susceptibility to reduction, certain generalizations on a class basis appear warranted. Quinonimide derivatives and numerous azo dyes are reduced very easily. On the other hand, the phthaleins (eosines, rhodamines and violamines) are relatively resistant to reduction. With a mixture of a dye of the first group and one of the second the selection of a suitable solvent in which only the first-group component will undergo reduction involves little difficulty. The results of six analyses of such mixtures (series 3 to 8) are recorded in the table. Triphenylmethane derivatives have characteristics intermediate between those of the two foregoing groups, and it will probably be possible to analyze most mixtures of a triphenylmethane dye and one belonging to either of the other groups. Acid Fuchsine (C. I. 692) has been titrated successfully in the presence of Eosine (C. I. 768) and Violamine G (C. L. 759), Crystal Violet (C. I. 681) in the presence of Rhodamine B (C. I. 749), and Methylene Blue in the presence of Fuchsine (C. I. 677). On the other hand, preliminary attempts to titrate Brilliant Scarlet 3R (C. I. 185) and Printing Violet R in the presence of Acid Fuchsine have failed. The reduction of the Acid Fuchsine in these experiments has been so slight, however, that a suitable solvent will probably be found.

The preliminary investigation of azo dyes of various types has indicated a considerable diversity in characteristics. It appears probable that azo dyes of relatively simple type and stilbene derivatives, such as Chrysophenine (C. I. 365), may be titrated successfully in the presence of direct dyes derived from benzidine and its homologs. With the only dye mixture of that sort which has been investigated the results obtained were not entirely conclusive. Duplicate titrations of 0.5 gm. samples of Brilliant Scarlet 3R in 20 per cent sulphuric acid gave values of 2.149, 2.148, 2.149 and 2.147 c.c. for consumption of normal titanous chloride solution. With mixtures of 0.5 gm. of Brilliant Scarlet 3R and 0.1 gm. of Sky Blue 5BX (C. I. 520) the values obtained were 2.159, 2.161, 2.160 and 2.158. A very slight degree of reduction of the Sky Blue is apparently indicated. The same discrepancy in results, however, was found in a number of further titrations, in which the reduction rate was retarded by increasing the acidity in some degree, by reducing the reaction temperature and by employing varying proportions of alcohol in the solvent. This may mean that some easily reducible impurity in the Sky Blue, rather than the Sky Blue itself, underwent reduction. In any case the discrepancy is not serious.

It is evident that the relative susceptibility of different dye classes to reduction by titanous chloride under different conditions has a diagnostic value in the identification of dyes. The practical utility of the phenomenon in this application will receive further investigation.

The data of successful titrations are recorded on page 417.

The dyes employed were of the ordinary textile grades. The titanous chloride solution was of 0.170 N. strength and of such acidity as to require 0.69 gm. of sodium hydroxide to neutralize 10 c.c. Under the acid conditions obtaining in many of the reductions only indirect methods of titration were possible, and the general practice was to run in a slight excess of titanous chloride and determine the excess by back-titration with ferric iron solution. Leuco Thiocarmine or leuco Methylene Blue was employed as an indicator unless the solutions already contained quinonimide dyes in reduced form. Indicator solutions are readily prepared by the exact reduction of acid aqueous solutions of the dyes with titanous chloride. They may be preserved under carbon dioxide and correction made for such slight oxidation as may occur between the intervals of their employment by adding a little more titanous chloride.

The analytical provisions recorded indicate merely the general principles upon which satisfactory analytical practice may be based rather than the optimum conditions which may be employed. In various instances it is probable that they may be modified with advantage in one respect or another.

Under the diverse conditions employed in the reductions, the titanous chloride consumption of various dyes differs slightly or even appreciably. Ponceau 6R, for example, requires about 2.5 per cent more titanous chloride for reduction under the conditions which permit of the simultaneous reduction of Eosine than it does under the conditions which inhibit the reduction of Eosine. Such differences do not invalidate the analytical results. Consistent values are obtained in duplicate runs with either practice, and the constant ratio between the two sets of values provides a reliable conversion factor.

The validity of the proposed methods is attested by the agreement in titration values of the more readily reducible component of the dye mixtures investigated when reduced alone and when reduced in the presence of the second component under conditions designed to prevent the reduction of the latter. In general, this agreement is satisfactory, and the conclusion is warranted that one component has been completely reduced and the other left intact. In most cases the apparent reduction of the first component is slightly incomplete in the dye mixtures, but this minor discrepancy obviously arises from the small excess of iron solution necessary to make the end point evident in the presence of the second dye. After the titrations recorded in the table had been made, it was found possible to eliminate this difficulty in large measure by adopting as the end point the value midway between the point of appearance of the distinctive color of the indicator upon oxidation and the point of its disappearance upon reduction. Alternate additions of minimum quantities of titanous chloride and iron solutions may be repeated until a thorough check is obtained.

The general practical utility of the methods is restricted in some degree, with the technic outlined, by the difficulty which may be encountered in the visual detection of end

TABLE OF ANALYTICAL DATA

Series	Dye Sample	Water cc.	Alc. cc.	Acetic Acid cc.	Sulfuric Acid cc.	25% Tartrate cc.	Temperature	Indicator Added	N. $TiCl_3$ Consumed cc.
1	0.3 gm. Methylene Blue (C. I. 922)	134		66			cold	none	1.519, 1.519, 1.518
	0.3 gm. Safranine O (C. I. 841)	134		66			cold	none	1.178, 1.180
	0.3 gm. Meth. Blue)								(1.513, 1.510, 1.514
	0.3 gm. Safranine)	134		66			cold	none	(1.184, 1.185, 1.182
2	0.5 gm. Printing Violet R (C. I. 888)	100		50			cold	none	1.147, 1.148
	0.1 gm. Thiocarmine (C. I. 928)	100		50			cold	none	0.234, 0.235
	0.5 gm. Printing Violet R)								(1.153, 1.149
	0.1 gm. Thiocarmine)	100		50			cold	none	(0.229, 0.232
3	0.5 gm. Ponceau 6 R (C. I. 186)	50	50		25		hot	Leuco Thiocarmine	1.827, 1.827
	0.5 gm. Ponceau 6 R)								
	0.5 gm. Eosine (C. I. 768))	50	50		25		hot	Leuco Thiocarmine	1.826, 1.825
	0.5 gm. Ponceau 6 R	50	50			50	hot	Leuco Thiocarmine	1.870, 1.873
	0.5 gm. Eosine	50	50			50	hot	Leuco Thiocarmine	1.645, 1.643
	0.5 gm. Ponceau 6 R)								
	0.5 gm. Eosine)	50	50			50	hot	Leuco Thiocarmine	3.516, 3.513
4	0.5 gm. Metanil Yellow (C. I. 138)	100			25		hot	Leuco Thiocarmine	3.496, 3.490
	0.5 gm. Metanil Yellow)								
	0.5 gm. Uranine (C. I. 766))	100			25		hot	Leuco Thiocarmine	3.500, 3.495
	0.5 gm. Metanil Yellow	100				50	hot	Leuco Thiocarmine	3.550, 3.554
	0.5 gm. Uranine	100				50	hot	Leuco Thiocarmine	1.828, 1.827
	0.5 gm. Metanil Yellow)								
	0.5 gm. Uranine)	100				50	hot	Leuco Thiocarmine	5.380, 5.378
5	0.5 gm. Ruby G (C. I. 179)	50	50		25		warm	Leuco Methylene Blue	2.187, 2.188
	0.5 gm. Ruby G))								
	0.5 gm. Rhodamine B (C. I. 749)	50	50		25		warm	Leuco Methylene Blue	2.189, 2.187
	0.5 gm. Ruby G	50	50			50	hot	Leuco Methylene Blue	2.245, 2.244
	0.5 gm. Rhodamine B	50	50			50	hot	Leuco Methylene Blue	0.445, 0.443
	0.5 gm. Ruby G)								
	0.5 gm. Rhodamine G)	50	50			50	hot	Leuco Methylene Blue	2.688, 2.690
6	0.5 gm. New Blue D A (C. I. 909)	50	50		25		cold	none	1.902, 1.902
	0.5 gm. New Blue D A)								
	0.5 gm. Rhodamine G (C. I. 750))	50	50		25		cold	none	1.900, 1.898
	0.5 gm. New Blue D A	50	100			50	hot	(direct titration)	2.010, 2.016
	0.5 gm. Rhodamine G	50	100			50	hot	(direct titration)	1.022
	0.5 gm. New Blue D A)								
	0.5 gm. Rhodamine G)	50	100			50	hot	(direct titration)	3.037
7	0.5 gm. Fast Wool Blue B (C. I. 209)	100		50			cold	Leuco Thiocarmine	1.806, 1.800
	0.5 gm. Fast Wool Blue B)								
	0.5 gm. Violamine G (C. I. 759))	100		50			cold	Leuco Thiocarmine	1.799, 1.803
	0.5 gm. Fast Wool Blue B	50	50			50	hot	Leuco Thiocarmine	1.799, 1.798
	0.5 gm. Violamine G	50	50			50	hot	Leuco Thiocarmine	1.514
	0.5 gm. Fast Wool Blue B)								
	0.5 gm. Violamine G)	50	50			50	hot	Leuco Thiocarmine	3.311
8	0.25 gm. Thiocarmine	100		50			warm	none	0.585, 0.587
	0.25 gm. Thiocarmine)								
	0.1 gm. Violamine B (C. I. 757))	100		50			warm	none	0.579, 0.582
9	0.5 gm. Acid Fuchsine (C. I. 692)	150					hot	Leuco Thiocarmine	1.771, 1.771
	0.5 gm. Acid Fuchsine)								
	0.2 gm. Eosine)	150					hot	Leuco Thiocarmine	1.767, 1.770
10	0.5 gm. Acid Fuchsine	150					warm	Leuco Thiocarmine	1.774, 1.773
	0.5 gm. Acid Fuchsine)								
	0.1 gm. Violamine G)	150					warm	Leuco Thiocarmine	1.769, 1.766
	0.5 gm. Acid Fuchsine	50	50			50	hot	Leuco Thiocarmine	1.722, 1.725
11	0.5 gm. Crystal Violet (C. I. 681)	150				5	hot	Leuco Methylene Blue	1.846, 1.843
	0.5 gm. Crystal Violet)								
	0.1 gm. Rhodamine G)	150				5	hot	Leuco Methylene Blue	1.837, 1.840

points with some dye mixtures. It is evident, however, that their scope may be extended by the application of electrometric methods of titration.[5]

In conclusion, it may be pointed out that it is sometimes possible to base a comparatively accurate calculation of the percentages of the component dyes in a mixture upon the value obtained for their total consumption of titanous chloride and a second value for total dye content by "difference," provided the unit consumption of titanous chloride of the two dyes in question is widely different.

The following general formula is applicable,

$$x = \frac{a - bc}{d}, \text{ where,}$$

(x) = gm. of the first component, or the dye with the greater unit consumption of titanous chloride.

(a) = the (total) consumption of normal titanous chloride solution by the mixture.

(b) = gm. of total dyes present (as obtained by an independent determination of total coloring matter by difference)

(c) = the titanous chloride consumption of one gram of the (pure) second component.

(d) = the difference in the consumptions of titanous chloride by one gram of the (pure) first and second components.

If this formula is applied to a mixture of the permitted food dyes, Naphthol Yellow S (C. I. 10) and Light Green SF Yellowish (C. I. 670), it will be found that an error of 0.03 c.c. of normal titanous chloride solution in the titration of a 0.2 gm. sample of the dye mixture introduces an error of less than 0.5 per cent in the calculation of the dye percentage. The probable error which might arise from inaccuracy in the determination of total dye content by difference would also be small. Although this method of analysis will have but limited practical utility in general dye analysis, it may occasionally prove of decided service in the case of mixtures of dyes with which the independent reduction of a single component is impracticable.

[5] Jones and Lee, J. Ind. Eng. Chem., **14** (1922), 46.

Oxycellulose and Its Tests

Nature of the Tests—The Two Types of Oxycellu lose—Test Results with Oxycellulose—The Older Qualitative Tests—Practical Interpretation

OXYCELLULOSES, or the products of oxidation of cellulose, have been found to fall between two extreme types having very different properties. Which type of oxycellulose may be formed in a given bleach depends on the acidity or alkalinity of the hypochlorite solution. On this account, it has been found that tests for oxycellulose must be carefully selected, and the results interpreted with these possibilities in mind. Critical studies of these phenomena, including the common tests, form the subject of an important paper in the Journal of the Textile Institute (1925, page T 13), whose more useful conclusions are here presented.

The term oxycellulose is uses as denoting a cotton cellulose whose chemical properties have been altered, however slightly, by the action of oxidizing agents. Many of the oxycelluloses on which previous scientific results have been reported have been complicated mixtures of uncertain significance. In the present work the more practical method was adopted of studying the progressive changes produced by oxidizing agents of increasing strength, but such that the form of the cotton was not destroyed.

The oxidations were carried out so that known amounts of oxygen were absorbed, the quantities ranging from 0.032 per cent of the cotton to 0.32 per cent. A better idea of these amounts can be obtained from the fact that a normal cotton bleach represents a consumption of 0.01 to 0.02 per cent oxygen. Further tests were made with a constant strength of hypochlorite, corresponding to 0.32 per cent oxygen consumption, and a range of hydrogen ion concentrations from acid through neutral to alkaline.

The various tests covered chlorine water, acid permanganate, hypochlorite and hypobromite. Enough experiments were run to give conclusions on oxidation in general, and on the effects of hypochlorites in particular. The resulting celluloses were characterized by a series of tests: copper number, Methylene Blue absorption, loss of weight on alkali boiling, ash alkalinity, and viscosity of cuprammonium solution, and were found to be of two distinct types.

The results in the series of varying hydrogen ion concentration are peculiarly interesting in that they may explain anomalies in previous work. The acid and alkaline concentrations are very slight, however, and difficult to maintain except by buffering, ranging from N/100,000 hydrogen ion to N/100,000 hydroxyl ion. The first gives an oxycellulose of relatively very high copper number and low Methylene Blue absorption, and the second a very low copper number and high Methylene Blue absorption, these being the typi-

cal characteristics of the two types of oxycellulose. Either test would detect oxycellulose produced at or very near the neutral point, but for that formed under very slightly acid or alkaline conditions either test alone might lead to a wrong conclusion.

The viscosity of its solution in "cuprammonium" is a widely recognized test for modified cellulose and follows closely parallel to tendering. Results show that, except in the earlier stages of oxidation, the two types of oxycellulose give the same viscosity changes, indicating that they are equally harmful to cellulose.

The tests available for oxycellulose are of two types. The first group, quantitative in nature, is more or less familiar and comprises the tests already listed, which have recently had special study by the British Cotton Industry Research Association. The second group consists of qualitative tests that have been in more common use for the detection of oxycellulose. The classification into two types recognized by the quantitative tests is only empirical, for there are all gradations between the two extremes.

The Two Types of Oxycellulose

The first is a reducing type of oxycellulose and has: High copper number and low Methylene Blue absorption; relatively high loss, on boiling with dilute caustic alkali.

The second has: Low copper number and high Methylene blue absorption; high ash alkalinity.

Both types have lowered viscosity.

The tests that have been used can be found discussed in greater detail in the references that follow.

Copper number is familar, but some confusion has resulted from differences in the proposed methods for its determination. In this work, Braidy's method was used, as recently described in detail in J. T. I. 15, T27 (1924).

Methylene Blue absorption was carried out in a definite way, as developed in J. T. I. 14, T297 (1923).

The loss of weight on alkali boiling was found by direct weighing. About 2 grams of the oxycellulose, dried and accurately weighed, was boiled for four hours with 200 c.c. of N/4 sodium hydroxide (equal to 1 per cent solution), then washed, soured, etc., and weighed again, the loss being determined by difference.

Ash alkalinity followed the method of J. T. I. 14, T279 (1923).

The viscosity of the oxidized cellulose in Cuprammonium was determined as in J. T. I. 15, T157 (1924).

Test Results with Oxycellulose

No case of oxidation of cellulose has been detected unaccompanied by a rise of copper number, so this measurement is considered a very valuable method for the detection of oxycellulose. However, when an oxycellulose has been boiled with dilute alkali, its copper number is reduced and may reach as low a value as that of normal unmodified cellulose. It may happen, therefore, that this test alone will fail to disclose the presence of oxidizing attack.

Results may be erroneous even when there has been no subsequent alkali boil, owing to the fact that different oxidizing agents, or the same oxidizing agents in solutions of differing acidity or alkalinity, produce oxycelluloses of very different copper numbers. This is true even where the extent of oxidation as measured by the consumption of oxygen in the same; a copper number of 0.4, for instance, may be produced by very slight oxidation, resulting in little loss of tensile strength, or by a severe oxidizing attack, with great loss of strength, just depending on the conditions of oxidation.

These facts are of special importance in relation to the detection of oxycellulose that has resulted from the reaction of hypochlorites on cotton, for in nearly neutral solutions very slight changes in acidity or alkalinity have a great effect upon the copper number. Fresh bleach liquors are nearly always slightly alkaline, but the products of the normal oxidation during bleaching—carbon dioxide and organic acids—frequently produce a neutral or slightly acid liquor.

The test by Methylene Blue absorption is of particular value when taken in conjunction with the copper number, the two tests together having far greater significance than either one alone. It is not a sensitive tests for very slight oxidation, but when oxycelluloses are boiled with alkali the Methylene Blue absorption remains substantially unaltered, and to this extent the test is superior to copper number for detecting oxidation.

Viscosity in cuprammonium solution is a sensitive indication of oxidizing attack, irrespective of the actual presence of oxycellulose. The fall of viscosity is approximately determined by the consumption of oxygen, and although alkali boiling reduces a high copper number resulting from oxycellulose, it does not restore the original viscosity. This measurement is most sensitive in the early stages of oxidation, and normal technical chemicking is almost invariably accompanied by a fall of viscosity, as well as by an increase in copper number. Two chemics separated by an alkali boil might result in no higher copper number than a single chemic, owing to the effect of the alkali, but there would be a proportionately greater effect on the viscosity. It is the most generally useful test to detect the effects of oxidation, but must be used with discretion, as it also responds to attack by acids or even by excessive alkali boiling.

Loss of weight on boiling with a 1 per cent caustic solution may be used as a substitute for the measurement of the copper number. No useful indication can

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Dyeing Acetate Silk

Part VIII

(*Continued from page* 410)

Dyeing Acetate Silk by Colloid Precipitation—Dyeing Acetate Silk with Various Bases and Developed or "Ice" Colors—The Application of Fast Bases

By CHAS. E. MULLIN, M. Sc., F. A. I. C.

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ANOTHER unusual method of dyeing acetate silk is that of Harrison, Burgess, and Ledward & Co., who obtained British Patent No. 179,384, March 21, 1921, covering a novel process which apparently has not come into general use. In this process the acetate silk is dyed with a colloidal dye solution prepared by adding to a solution of the dye or its leuco compound a protective colloid, such as gelatine, casein, saponin, starch, or their hydrolytic products; followed by a precipitant (except metallic chlorides), preferably a precipitant which is absorbed by acetate silk. Suitable precipitants are: For basic dyes, a weak alkali such as ammonia; a salt of an acid metallic oxide, such as molybdates, tungstates or stannates, or an organic compound of acid character such as tannic acid or salts of phenols, naphthols, aromatic acids, hydroxy acids, sulphonic acids, etc.; for acid or direct cotton dyes, a salt of an organic base such as aniline, benzidine, dianisidine, phenylenediamine, naphthylamine, etc.

Sulphur dyes are dissolved in sodium sulphide, hydrosulphite, etc., and vat dyes in sodium hydrosulphite, the same precipitants being used as in the case of direct dyes. Mixed goods containing cotton may be dyed with sulphur or vat dyes as described above, the cotton threads being subsequently dyed with direct dyes. The provisional specification describes also the use of salts of aluminum, barium, magnesium, etc., as precipitants for direct or acid dyes, and of oxidizing agents such as air, or metal salts as precipitants for vat or sulphur dyes; it also refers to the use of alizarine dyes using metallic salts, mordants or organic substances as precipitants.

Method No. 61: As an example, one hundred grams of acetate silk are worked for five minutes in a bath containing 2 g. of indigo, 8 g. caustic soda, 5 g. sodium hydrosulphite, 2 g. of glue, and 2 liters of water at 60 to 70 deg. Cent. (140 to 157 deg. Fahr); a solution of 0.5 g. dianisidine in 4 c.c. of glacial acetic acid and 200 c.c. of water is then added in successive small quantities. The dyed fiber is removed from the bath and oxidized in the air. This process is more satisfactory for vat and sulphur dyes than with the direct cotton dyes, as the latter are not fast to soap and alkalies when applied by this method.

Method No. 62: The example for basic dyes states that 100 g. of acetate silk may be dyed in a 2-liter bath containing 1 g. of Magenta or 2 g. of Methylene Blue and 2 g. of glue, raising the temperature to 70 to 80 deg. Cent. (158 to 176 deg. Fahr.). A solution of 2 g. of sodium stannate in 200 c.c. of water is then added gradually, while working the fiber, saponin may replace the glue, and tannic acid the stannate. Evidently this method applies only to Celanese as Lustron has such a high affinity for the basic dyes that this method is hardly necessary to dye it. Diamine Sky Blue and other direct cotton colors are applied in the same manner, only adding 0.5 g. of dianisidine base in 2 c.c. of glacial acetic acid in 200 c.c. of water, in place of the sodium stannate solution.

The foregoing methods, patents, etc., more or less cover the methods of applying the older and better known commercial dyes to acetate silk, and we are now ready to consider the application of certain bases, water insoluble products and Ionamines to acetate silk by special variations of the older methods or by the new methods which have been developed particularly for dyeing this fiber. As the ice or developed colors are the oldest of this group, they will be considered first.

Dyeing Acetate Silk with Various Bases and "Ice" or Developed Colors

Acetate silk has a distinct affinity for the many organic bases such as dianisidine, *a*-naphthylamine, benzidine, etc., and developed colors may be formed or the fiber by diazotizing and developing these bases. These dyes are not difficult to apply and produce a wide range of shades of excellent fastness to washing. As the affinity of acetate silk for the naphthols is only slight, the application of the developed colors to this fiber differs from their application to cotton, where the cotton is padded with the naphthol solution first. In dyeing acetate silk with these products, the acetate silk is first treated with the aromatic amine or diamine, and this is then diazotized on the fiber and developed.

The bases may be applied from a solution of their hydrochlorides, but in many cases quite a different shade and an increased affinity of the base for the acetate silk is obtained when the free base is used instead of its hydrochloride. This increased affinity is sufficient to produce blacks on developing such bases as benzidine,

toluidine, *p-p*-diaminodiphenylamine, etc., even when dyed from a cold bath. The free bases may be applied by dissolving the soluble hydrochlorides of the bases in water and adding sufficient alkali to neutralize the bath; or the free base may be dissolved in a suitable dispersing agent, as in the Dispersol method of dyeing acetate silk, which will be discussed later. Even such insoluble dyestuffs as Induline, Nigrosine, Rosaniline base, Methyl Violet base and Spirit Red III may be applied to acetate silk in fine suspensions by pouring their alcoholic solution into water. It is noticeable that Spirit Red III in alcoholic solution has no affinity for acetate silk, while the aqueous suspension dyes the fiber. However, alcohol is possibly not the best solvent for obtaining this effect, which will be considered more fully under the Dispersol dyes.

The foregoing statements regarding the bases cover the whole development of the new dyes for acetate silk. As we go further into the matter, we will see that many of these bases may be, and what is more, really are applied to the fiber by a variety of these more or less special methods. Therefore, the same base may be the principal dyeing constituent of quite a few of the many brands or varieties of special dyes for acetate silk. Practically all of these new dyes for acetate silk are either bases, azo compounds, amines or vat dyes of a particular character, etc. This refers not only to the Azoniles, Acedronoles, Silkons, Azoics, Azoles, and Developed Azonines, but the Ionamines and Cellits, as well as the dispersol dyes, which include the S. R. A., Duranols, Celatenes and Direct Azonines. In the Azoniles, Acedronoles, Silkons, Azoics, Azoles and Developed Azonines, the bases are dissolved by the aid of hydrochloric acid. In the Ionamines the bases or other insoluble compounds are solubilized by the introduction of the omega sulphonic group, which is external to the dye nucleus, and therefore splits off in dyeing. The Cellit dyes are no doubt insoluble bases, etc., solubilized by means of bisulphite, which splits off in the dyebath. In the dispersol paste dyes, the insoluble compounds are dissolved in a suitable water soluble solvent, usually organic in nature, and are possibly precipitated in a colloidal condition on dilution in the dye bath. The S. R. A.'s, Duranols, Celatenes and Direct Azonines are pastes of this character.

One of the earliest patented methods of dyeing acetate silk was German Patents No. 198,008 and No. 199,559, February 19, 1907, to Knoll & Co.; French Patent No. 383,636, November 6, 1907; British Patent No. 24,284, November 2, 1907, and United States Patent No. 961,241, June 14, 1910, also cover this process. In these patents it was suggested to dye acetate silk with developed colors by the aid of solvents or swelling agents; these patents were discussed under Method No. 53, in connection with the solvent methods of dyeing. United States Patent No. 979,966, December 27, 1910, to E. Knoevenagel, covers the use of aqueous solutions of phenols to dye acetate silk, and coupling with diazonium salts is suggested. In United States Patent No. 1,002,408, September 5, 1911, the last named inventor suggests treating the fiber with an aqueous solution of an aromatic amine, as for instance aniline, *p*-nitraniline, *a*-naphthylamine, benzidine or *p*-aminophenol, to constitute one component of a dye formed on the fiber.

It therefore appears that while this property of the bases to dye acetate silk is not a new discovery, it has only come into general use recently. When applied in this manner aminoazobenzene gives an excellent yellow, which upon diazotization and development with *m*-phenylenediamine becomes brown; with *m*-toluylenediamine a redder shade; with naphthol a scarlet; with resorcinol an orange. Benzidine on the fiber and coupled with *m*-phenylmediamine gives rich browns. Orthoanisidine and chloranisidine may be coupled with *a* or *B*-naphthol, *m*-phenylenediamine or resorcinol to give good shades. Dichlorobenzidene coupled with *B*-naphthol gives a fine crimson; with *m*-phenylenediamine a browner shade, and with chloranisidine a rich orange. Para-amidodiphenylamine when oxidized on the fiber gives a splendid black (Diphenyl Black), and no doubt many other oxidizable bases may be used in the same manner upon acetate silk. Para-red may be developed on the fiber with *p*-nitraniline, as given under Method No. 53; and according to British Patent No. 211,720 which will be considered under the dispersol dyes, certain aminoanthraquinones in fine suspension or colloidal solution may be applied to acetate silk. These products are also used in the preparation of Ionamine Blue R and G, which will be discussed with the other Ionamines.

Claven in British Patent No. 187,964, October 27, 1921, covers the production of azo or ice colors on acetate silk by adding soluble salts to dye baths containing the parent amine or the developer or to both. Chlorides of ammonium, sodium, potassium, barium, calcium, magnesium zinc or tin, or sulphates of sodium, potassium or magnesium are given as suitable salts. Also see British Patents No. 176,535, 182,830, 187,964 199,754 and 204,179. Protective colloids, such as gelatine, silk boil-off liquor, Turkey Red oils, albumin, tannates soaps, etc., may also be added when the bases or developers are used in neutral or alkaline baths. These assistants allow the use of baths of lower alkalinity and give a more even absorption of the color constituent.

Method No. 63 For example, 1 kg. of acetate silk may be dyed black by soaking it for 30 to 45 minutes at 60 deg. Cent. (140 deg. Fahr.) in 20 to 25 liters of a solution containing 25 g. of dianisidine hydrochloride, 50 g. of magnesium chloride, and 40 g. of sodium bicarbonate, and washing. It is diazotized in a bath containing 50 g. sodium nitrite and 200 c.c. of concentrated hydrochloric acid for 30 minutes at 15 deg. Cent. (59 deg. Fahr.) and developed in a 20 or 25 liter bath containing 20 g. of *a*-naphthylamine hydrochloride, 8 g. of sodium bicarbonate and 50 g. of magnesium chloride for 1 hour at 65 deg. Cent. (149 deg. Fahr.). It is then washed, diazotized as before, and soaked for an hour at 65 deg. Cent. (149 deg. Fahr.) in a slightly alkaline bath containing 20 l. of water, 5 l. of silk boil-off liquor, 30 g. of 5-hydroxy-*a*-naphthylamine, and 50 g. of magnesium

chloride, whereby the black shade is developed. A final soaping at 60 deg. Cent. (140 deg. Fahr.) is given.

Acetate silk may be dyed with the ice-colors in a bath containing protective colloids, according to British Patent No. 199,754, January 5, 1922, to Clavel. He suggests that (*a*) the base and developer may be applied in the same bath and then diazotized in a second bath; or (*b*) the base may be applied and diazotized in the one bath, developing in the second bath; or (*c*) the base may be applied in one bath, and then diazotized and developed in the second bath, in the presence of a suitable protective colloid, such as gelatine, silk boil-off liquor, etc.

Method No. 64 For example, one kilogram of acetate silk may be dyed a brown-red shade by working it for a half hour at room temperature in a bath containing 25 liters of water, 20 g. aminoazobenzene, 30 g. *B*-naphthol, 5 g. of Capri Blue (for shading) and 2 l. of silk boil-off liquor; afterward adding 5 g. of magnesium chloride and working for one-half hour at 60 deg. Cent. (140 deg. Fahr.). It is then washed, diazotized cold in a bath containing 20 l. of water, 50 g. sodium nitrite and 100 c.c. of concentrated hydrochloric acid.

Method No. 65: Another example specifies a 25 l. bath containing 20 g. aminoazo-toluene and 2 l. of silk boil-off liquor. After working for 30 minutes, 50 c.c. of sulphuric acid and 50 g. of sodium nitrite are added. Wash and develop in a 60 deg. Cent. (140 deg. Fahr.) bath containing 25 l. of water, 30 g. of *B*-Naphthol and 50 g. of soap.

Method No. 66: More recently in British Patent No. 204,179, July 28, 1922, Clavel states that in the production of azo dyes upon acetate silk the development may be effected at temperature of from 60 deg. Cent. (140 deg. Fahr.) to boiling and the application of the bases may be effected at the same high temperature or not. The bases and developers may be applied in separate baths or in a combined bath, and the diazotization is effected in a separate bath at ordinary or low temperature. Readily hydrolyzible salts of strong bases, such as sodium acetate or free bases such as caustic soda, may be added to the base baths and acids or acid salts,, e. g., hydrochloric or acetic acids or sodium hydrogen phosphate may be added to the developing baths. Protective colloids such as gelatine, silk boil-off liquor or sulphonated fatty acid soaps may also be added, and soluble salts such as chlorides of magnesium, ammonium, zinc or tin may also be added to the baths.

Ledward & Co., Burgess and W. Harrison obtained British Patent No. 193,646, February 4, 1922, on a single bath process of forming azo dyes on acetate silk. In this process the silk is steeped in a dye bath containing the amino or/and phenolic components of the dyestuff, subsequently adding sodium nitrite and then acid, whereby the color is developed. Temperatures between 30 and 80 deg. Cent. (86 to 176 deg. Fahr.) are used, and when the resulting dye tends to form a precipitate in the bath, a protective colloid, such as gelatine or starch, is added. The colors may be topped with basic or other dyes.

Method No. 67: For example, a red color is obtained on 100 parts of acetate silk by treating it at 60 deg. Cent. (140 deg. Fahr.) in a dye bath of 4,000 parts water, containing 2 parts of aminoazobenzene dissolved in 2 parts of hydrochloric acid. After 30 minutes 10 to 20 parts of sodium acetate is added to aid the exhaustion, and after a further 30 minutes, 2 parts of glue dissolved in water, and 2 parts of *B*-hydroxynaphthoic acid dissolved in water containing 2 parts of ammonia. After a further 15 or 20 minutes, 0.7 parts of sodium nitrite and 10 parts of formic acid are added, the temperature being maintained at 60 deg. Cent. (140 deg. Fahr.). Finally a further 0.7 part or more of sodium nitrite is added, whereby the silk develops a full red shade, which may be topped with Butter Yellow or Nile Blue.

Method No. 68: A navy blue on 100 parts of acetate silk may be obtained by treating it in a bath containing 2 parts of dianisidine, 2 parts of 36 deg. Tw. hydrochloric acid and 4,000 parts of water at 50 to 70 deg. Cent. (122 to 158 deg. Fahr.) for 15 minutes. Ten parts of sodium acetate are then added and the treatment continued for 15 minutes. Two parts of glue dissolved in hot water and 4 parts of *B*-hydroxynaphthoic acid dissolved in 20 parts of hot water containing 2 parts of 0.88 Sp. Gr. ammonia are added, and in 15 to 20 minutes 1.2 parts of sodium nitrite in 4 parts of water and 15 parts of 90 per cent formic acid are introduced. After working for 30 minutes, 1.2 parts more of sodium nitrite are added and the treatment continued for another 30 minutes. A black shade is obtained from 1-aminobenzene-4-azodimethylaniline and *B*-hydroxynaphthoic acid. The dyeings may be made purer and clearer by an after-treatment with reducing agents, such as formaldehyde-hydrosulphite or stannous chloride in acid solution.

Method No. 69: The recent French Patent No. 25,785 covers the production of black on acetate silk by an unusual process. In the example, 1,000 grams of acetate silk are treated for from 30 to 45 minutes in a 25-liter bath containing 20 g. dianisidine salt, 50 g. magnesium chloride, and 40 g. of sodium bicarbonate The fiber is then washed, first in soft water followed with hard water and diazotized in a 20-liter bath containing 50 g. of sodium nitrite and 200 g. of hydrochloric acid. It is developed for one hour in a bath containing 20 g. of naphthylamine chlorohydrate and 8 g. of sodium bicarbonate, after which it is washed and then again placed in the developing bath. It is then washed again and treated for an hour in a 20-liter bath containing 5 liters of filtered soap solution, 35 g. aminonaphthol (gamma acid) and 50 g. of magnesium chloride, to give a deep black color. A final washing with water at 60 deg. Cent. (140 deg. Fahr.) followed by a soap washing concludes the treatment. The color may be given a brightening treatment if desired.

United States Patent No. 1,498,315, June 17, 1924, to J. Baddiley and J. Hill, covers the same process as British Patent No. 202,157, discussed on page 316 of part 5 of this series. This patent states that acetate silk may be dyed directly with soluble amino azo dyes containing one or more carboxyl groups and also containing a diazo-

tizable amino group, so that after dyeing the direct shade with the carboxylated azoamino dye, different shades may be obtained by diazotization and development of the fiber. The dyes are applied directly as their sodium salts in an aqueous dye bath, either alone or with sodium chloride, sulphate or a dilute acid to assist exhaustion.

The following examples of acetate silk dyes are given with the colors obtained directly and by diazotization and development: When *m*-aminobenzoic acid with *o*-anisidine is dyed directly, a yellow color is obtained which upon development with *B*-hydroxynaphthoic acid gives a magenta red color; or with *p*-aminodiphenylamine a golden yellow color. *m*-Aminobenzoic acid with anthranilic acid gives a direct yellow color which with *B*-naphthol develops a red; with *B*-hydroxynaphthoic acid a bluish-red; or with *p*-aminodiphenylamine an olive color. *p*-Aminosalicylic acid with *a*-naphthylamine also gives a direct yellow which with *B*-naphthol gives a violet; *B*-hydroxynaphthoic acid a reddish-blue; or with *p*-aminodiphenylamine an olive color.

5-Acetylamino-2-amino-4-methoxytoluene with *B*-hydroxynaphthoic acid gives a direct bluish violet color which upon diazotization and development with *B*-naphthol gives a greenish blue; with *B*-hydroxynaphthoic acid a blue; or with *p*-aminodiphenlyamine a reddish-violet color. *m*-Aminobenzoic acid with *p*-xylidine and *m*-phenylenediamine gives a direct red shade which may be developed with *B*-naphthol or *B*-hydroxynaphthoic acid to a reddish-brown or with *p*-aminodiphenylamine to give a brown color. *m*-Aminozenboic acid with *p*-xylidine and *a*-naphthylamine, when dyed directly gives a brownish-red, which upon development with *B*-naphthol is a reddish-violet; or with *p*-aminodiphenylamine a brown color. *m*-Aminobenzoic acid with *m*-toluidene gives a direct yellow shade, and when developed with *B*-naphthol a scarlet; or with *B*-hydroxynaphthoic acid a blueish-red color.

Anthranilic acid with anisidine dyes a direct orange, which changes to a bluish-red upon development with *B*-naphthol; or with *B*-hydroxynaphthoic acid a reddish-blue color. *m*-Aminobenzoic acid with *a*-naphthylamine dyes a direct reddish-orange, which develops to a reddish-violet with *B*-hydroxynaphthoic acid. *p*-Aminobenzoic acid with aminohydroquinonedimethyl either dyes acetate silk an orange color which develops reddish-violet with *B*-naphthol; or a blue with *B*-hydroxynaphthoic acid. *m*-Aminobenzoic acid with 4-nitro-2-anisidine dyes a direct greenish-yellow, which with B-naphthol develops a reddish-orange; or with *B*-hydroxynaphthoic acid a red color. *m*-Aminobenzoic acid with 1, 2-aminonaphthol either dyes a direct, red which develops a greenish-blue with *B*-naphthol; or a bluish-green with *B*-hydroxynaphthoic acid.

The Application of the Fast Bases

From certain experiments made by the writer, it would appear that certain fast bases such as are marketed by Griesheim Elektron Company may be applicable to acetate silk under the proper conditions. These bases are marketed as Fast Orange R Base (*m*-nitraniline), Fast Garnet B Base (*a*-naphthylamine), Fast Garnet G Base (*o*-aminoazotoluene), Fast Red B Base (5-nitro-2-amino anisole), Fast Red BB Base (*o*-anisidine), Fast Red G Base (3-nitro-4-aminotoluene), Fast Red GL Base (3-nitro-4-aminotoluene), Fast Red 3 GL Base and Fast Red 3GL Base Special (2-nitro-4-chloroaniline), Fast Red R Base (4-chloro-2-aminoanisole), Fast Red RL Base (5-nitro-2-aminotoluene), Fast Scarlet G Base (4 nitro-2-aminotoluene), Fast Scarlet R Base (4-nitro-2-aminoanisole), Fast Blue B Base (dianisidine), Fast Black LB Base (2-ethoxy-benzeneazo-*a*-naphthylamine) and Fast Yellow G Base (*o*-chloroaniline). No doubt some of these bases can also be applied to acetate silk by the dispersol method of dyeing which will be discussed later.

These bases may then be coupled with certain naphthols, which in some instances also have a certain affinity for acetate silk. The following are recommended for use with the bases just mentioned. Naphthol AS (*B*-hydroxynaphthoic acid anilide); Naphthol AS-BS, formerly called BS or AN (*B*-hydroxynaphthoic acid *m*-nitroanilide); Naphthol AS-RL (*B*-hydroxynaphthoic acid *m*-nitroanilide); Naphthol AS-RL (B-hydroxynaphthoic acid *p*-anisidide); Naphthol AS-SW (*B*-hydroxynaphthoic acid *B*-naphthalide), and Naphthol AS-G (diacetoacetotoluide). These products usually give very fast colors.

It is also possible that some of the same company's Rapid Fast dyes may be applicable for printing acetate silk. These Rapid Fast dyes are faintly alkaline mixtures of stabilized diazo compounds and various naphthols. Rapid Fast Red B is a mixture of the nitrosamine of diazotized 5-nitro-2-aminoanisole and Naphthol AS. Rapid Fast Red BB is a mixture of the same nitrosamine with Naphthol AS-BS. Rapid Fast Red GG is a mixture of the nitrosamine of diazotized *p*-nitroaniline and Naphthol AS. Rapid Fast Brown B powder is a mixture of Rapid Fast Red GG and an unidentified shading compound. Rapid Fast Red 3GL is a mixture of the nitrosamine of diazotized *o*-nitro-*p*-chloraniline and Naphthol AS. Rapid Fast Blue B powder is a mixture of Naphthol AS and probably tetrazotized dianisidine. Rapid Fast Red GZ is a mixture of stabilized diazotized 2, 4-dichloroaniline and Naphthol AS. Rapid Fast Orange RG is a mixture of nitrosamine of diazotized *o*-nitro aniline and Naphthol AS. Rapid Fast Red CL is an alkaline paste of Naphthol AS and the nitrosamine of diazotized *m*-nitro-*p*-toluidine.

F. M. Rowe [Journal of the Society of Dyers and Colourists, 40, 218-28 (1924)], gives tables showing the colors given by a wide variety of bases when coupled with the various naphthols, etc., and no doubt many of these could be used on acetate silk as well as on acetate silk unions under certain conditions to give a wide variety of very fast colors.

British Patent No. 214,112 was discussed under Vat Dyes, but the colors obtained directly by the hydrosulphite

vat may be diazotized and developed with *B*-Naphthol. British Patent No. 202,157, discussed under Acid Dyes and United States Patent No. 1,498,315 which covers the same process, specify the use of carboxylated amino-azo dyes which may be diazotized and developed on the fiber. British Patent No. 200,873, under the Ionamines and British Patent No. 202,157, under Acid and Mordant Dyes, also refer to developed colors.

Acetate silk may be dyed a pure yellow shade by means of tartrazine from a bath containing (*a*) 0.75 per cent of sodium dioxytartrate dissolved with just sufficient hydrochloric acid, and (*b*) 1.3 per cent of phenylhydrazine dissolved in acetic acid. The two solutions (*a* and *b*) are mixed in the dye bath and the acetate silk immediately entered. It is turned for ten minutes cold, the temperature gradually raised to 50 deg. Cent. (122 deg. Fahr.) and the dyeing continued for twenty minutes. Cotton present is not stained and the color on the acetate silk is fast to soaping at 40 to 50 deg. Cent. (104 to 122 deg. Fahr.). By substituting *p*-nitrophenylhydrazine for the phenylhydrazine, an orange color results. A terra-cotta shade is obtained in a similar manner, in a single bath containing "nitrosamine" and 1, 2, 4-toluylenediamine in the presence of acetic acid.

(Part IX will appear in an early issue.)

FRENCH DISTRIBUTION OF REPARATION DYES

The importation of dyes from Germany in the form of reparations in kind has passed from the hands of the Union Syndicate des Fabricants de Matieres Colorantes under the control of a subsidiary organization founded under the auspices of the Union, according to Consul G. F. Wadley, Paris.

This company, with the title of "Bureau Central d'Approvisionement en Matieres Colorantes de Prestation," has been formed as a limited liability co-operative society with a capital of 50,000 francs, and with offices in Paris. The statutes of the society provide that it has been formed for the purpose of ordering, for the account of shareholders and others, of importing, distributing and of paying the cost of dyes and intermediates to which the French Government is entitled as reparations in kind in virtue of the protocol of London on August 16, 1924.

The founders and administrators of the society have been chosen from among members of the boards of directors of the various dye producers and users belonging to the Union Syndicate des Fabricants de Matieres Colorantes.

At Trenton, N. J., the Rayon Processing & Warp Sizing Company, 297 Main Street, has been formed to process yarns and fabrics of all kinds, especially artificial silk, and in all ways handle and deal in silk, artificial silk, wool, and other textile fabrics. Capital stock, $50,000; par value, $100. Henry Smith, Charles W. Yenny and Veranda Cerce, all of Paterson, hold a total of ten shares.

METZ HEADS NEW DYESTUFF CONSOLIDATION

On July 1 a new concern known as the General Dyestuff Corporation, organized under the laws of the State of New York, takes over the entire dyestuff business, with all branch offices, of H. A. Metz & Co. In addition, the new corporation has acquired the business heretofore carried on by the Consolidated Color & Chemical Company and the Central Dyestuff & Chemical Company, together with their stock of dyestuffs, and in future will act as sole importer of the dyestuffs manufactured by Farbwerke vorm. Meister Lucius and Bruening of Hoechst.

B. A. Ludwig, formerly vice-president of the National Aniline & Chemical Company, who was recently appointed the sole importer of the products manufactured by Leopold Cassella & Co., of Frankfurt, joins the General Dyestuff Corporation as vice-president and director, and brings to it the agency for Cassella products. Herman A. Metz, president of H. A. Metz & Co., will also be president of the new corporation; Charles L. Gagnebin is named as first vice-president and Almuth C. Vandiver as secretary and treasurer.

The main offices of the General Dyestuff Corporation will be continued at 122 Hudson Street, New York, with branches in Boston, Providence, Philadelphia, Chicago, Charlotte, N. C., and San Francisco.

DISTRIBUTION OF COLOR CARDS IN JAPAN AND FOREIGN COUNTRIES

That the Standard Color Cards of America have been adopted by manufacturers, exporters and buyers throughout the Japanese market was learned recently by the Textile Color Card Association. This is substantiated by the marked increase in the distribution of color cards to firms in Japan and to their representatives abroad.

This demand has also brought about a similar action from many who carry on trade with Japan. How far reaching this effect is, may be illustrated by orders for cards received from Johannesburg, Melbourne and London, either from agents of Japanese firms or from importers dealing with these companies.

As well as the members in Japan, China and the Philippines, there are a growing number to be found in every important commercial center in the world. In 1921 the cards had a distribution in twenty-seven foreign countries. Since that time the demand has rapidly increased in England and the British Colonies, as in Japan. The circulation of cards has become more widely extended in all foreign countries.

The Textile Color Card Association in the standardization of colors has thus created a color language which is used with mutual understanding by all nations. Its benefits have been recognized and communications in its diction have been carried on wherever the "dialect of color" is spoken.

Proceedings of the American Association of Textile Chemists and Colorists

The American Association of Textile Chemists and Colorists

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ANNUAL MEETING OF THE NEW YORK SECTION

The annual meeting of the New York Section was held Friday, May 22, 1925, at the Newark Athletic Club, Newark, N. J. An excellent dinner was served at 7 P. M. and the meeting proper was opened by Chairman Edward F. L. Lotte at 8.45 P. M. About sixty members were present.

A Nominating Committee, consisting of Messrs. Hadley, Harold and Mitchell, which had been appointed at the previous meeting by Chairman Lotte, reported the following candidates for officers for the 1925-26 season:

For Chairman and Vice-Chairman—
O. R. Flynn
Herbert Grandage

For Secretary—
D. P. Knowland
Giles Low

For Treasurer—
J. J. Sokolinski
H. F. Wilmot

For members of Sectional Committee—
E. H. Killheffer
G. W. Knight
Ed F. L. Lotte
R. R. Sleeper
F. P. Summers
P. J. Wood

Printed ballots bearing the above names were distributed to the members present during the dinner, which tellers collected and tabulated before the opening of the meeting proper.

Directly after the meeting opened Chairman Lotte called for the report of the tellers and Mr. Herrmann, reporting for them, announced that the following officers had been elected for the coming season:

Chairman—
Herbert Grandage

Vice-Chairman—
O. R. Flynn

Secretary—
D. P. Knowland

Treasurer—
J. J. Sokolinski

Members of Sectional Committee—
E. H. Killheffer
Ed F. L. Lotte
P. J. Wood

Chairman Lotte—I am sure we are all glad of the officers we are going to have for the ensuing year. I am sure I have had great help from the Vice-Chairman, Mr. Grandage. He hasn't always been here but he has always been ready to help and I am sure came whenever he could. I want to thank the members as well for the support that has been given me during the past year. I

enjoyed the work and I feel sure that Mr. Grandage will enjoy it, too. As he has now been elected Chairman, I think it is his job to take charge of the meeting. (Applause.)

Chairman Grandage—Gentlemen, I appreciate the honor and thank you for it. I shall do the very best I can to be deserving of it.

A. P. Howes—Before proceeding with the meeting I think it would be most appropriate if the Section were to extend a rising vote of thanks to the retiring Chairman for the very efficient, painstaking and helpful manner in which he has conducted the affairs of the Section during the past year.

. . . A rising vote of thanks was given to Mr. Lotte. . . .

Chairman Grandage—I feel reasonably sure that all the members know the new officers for the coming year. Some of them know me. I am standing up now. I should like to invite the other officers elected to-night, who are here, to stand up also, so that you can all take a look at them.

We have with us to-night Dr. E. J. Rath, connected with the Grasselli Dyestuff Corporation, who is going to present the subject, "New Developments in Naphthol AS." I am sure we will be very interested to know what he has to say. Dr. Rath! (Applause.)

. . . Dr. Rath presented his prepared paper which follows:

New Developments in Naphthol AS

By Dr. E. J. Rath

Grasselli Dyestuff Corporation

It is a particular pleasure to me to talk to this American society of experts to-night about a subject which for years has occupied me from a scientific as well as a practical standpoint. Naphthol AS and its application for the preparation of azo colors on the fiber has during the last years made tremendous strides forward, and the improvements which were accomplished by scientific study of the dyeing process itself have interested ever-increasing circles of the cotton and allied industries.

Dr. Rose gave a lecture on this subject before your society at the occasion of your annual meeting in Philadelphia, printed in your Proceedings, in which he illustrated the material chiefly from the scientific standpoint and in which he emphasized the valuable research work which Grieshiem had accomplished in the direction of working out new representatives of this interesting group of dyeing components. In Germany we have been particularly pleased to read from his article the spirit of unbiased recognition toward our scientific progress. Permit me to-day to say a few words to you about the *coloristic* side of the problem. The coloristic work on the subject was naturally of the highest importance for the practical development in order to secure the fruits of science for the benefit of commerce.

While the purely scientific development brought about a considerable enlargement of the scale of shades, the coloristic work, of course, always in close conjunction with science, marched largely in three distinct directions: Fastness to light, fastness to crocking, and the adapting of the dyeing process to our modern dyeing machines. This demand could naturally not be wholly satisfied with the two old representatives of the group—Naphthol AS and AS-BS—the invention of which dates back to 1912 and the properties of which are certainly well known to you. Griesheim, together with Bayer, commands to-day a range of eight naphthols, which are all classified under the group name AS and which are differentiated from each other by additional letters. These are the following: Naphthol AS, Naphthol AS-BS, Naphthol AS-BO, Naphthol AS-RL, Naphthol AS-SW, Naphthol AS-BG, Naphthol AS-BR, and Naphthol AS-G.

Qualities of the Different Naphthols

Whereas the well-known Naphthol AS is practically the prototype of the group, giving with the sixteen different bases the shades from bright orange to scarlets, reds, Bordeaux, and blue, the Naphthol AS-BS, whose combinations are somewhat inferior to light but brighter and more bluish in shade, serves nearly exclusively as a shading naphthol in combination with AS.

The Naphthol AS-BO is the special product for producing fast-to-light Bordeaux shades, whereas the Naphthol AS-RL gives with almost all bases extremely fast-to-light dyeings, of which the most remarkable is the combination with Fast Red RL base; this latter is especially valuable for the production of a brilliant and fast-to-light pink which in this respect can be fully classed with the vat pinks.

Naphthol AS-SW, originally destined exclusively for the production of fast-to-bleaching black, with Fast Black LB Base can also, it had been found, successfully be employed for the preparation of a brilliant red which is fast to caustic boiling even under a slight pressure, and is therefore a welcome substitute for the Alizarine Red.

Naphthol AS-BG and AS-BR are special products for brown shades, the first giving a yellowish brown of most excellent fastness to light, the second a more reddish type.

Naphthol AS-G serves for the creation of yellow shades and these are the first azo compounds which stand kier boiling even under the highest pressure with highly concentrated caustic, a fact which is particularly remarkable. Outside of these differences in shades and fastness properties it is of interest to mention the differences which these eight naphthols show in their relative substantivity. At this occasion I assume a short excursion into the field of colloidal chemistry. An investigation of beta-naphthol and the different naphthols of the AS group showed that

a solution of beta-naphthol in caustic has the properties of a real solution, whereas the AS types give decidedly colloidal solutions. Furthermore a relation could be established between the degree of dispersion of these solutions and their substantivity to cotton in such a manner that the solutions of a higher degree of dispersion possess a rather inferior substantivity, whereas substantivity rises with the decreasing degree of dispersion. All of the naphthols of the AS type are more or less substantive products. That means they have other properties than beta-naphthol has. They go into the fiber but in a rather different manner from each other. The eight naphthols can practically be classified into three groups regarding their substantivity. Naphthols of a high degree of dispersion and inferior substantivity, classified according to the degree of dispersion, are AS-G, AS, AS-BS, and AS-RL. Next follow AS-BG and AS-BO, and the last group of a low degree of dispersion and a very high substantivity are AS-SW and AS-BR. This means that a bath of a naphthol of the first group exhausts only to an amount of about 20 per cent, from which a calculation can be deducted as to the amounts which are needed to bring successive baths to the original concentration. With increasing substantivity the exhaustion rises and reaches with AS-SW and BR about 80 per cent, which practically means a substantivity, as good as a well yielding direct dyestuff possesses.

Combining the Naphthols

These differences in substantivity allow the natural conclusion that only those naphthols can be combined with each other which belong to the same class in this respect. While therefore the naphthols can be combined under certain conditions, it is not advisable to go into the mixing of the bases since these differ all in their behavior as to their coupling energy; that means, that from a mixture the quick coupling component would exhaust faster than the slow one.

The differences in substantivity will furthermore influence the selection of the dyeing method. Naphthol AS and its less substantive relations are chiefly used for such processes where the material is only for a short time in contact with the dye liquor; that is, in piece dyeing on the padding machine and in dyeing warps in the continuous box machine. The substantive naphthols, however, find their employ in all such cases where the cotton is in longer contact with the liquor, that is in dyeing skeins on the open tub, loose cotton in the packing system, beams, cops or packages in the different systems of dyeing machines, warps in the Scotch tub or piece goods on the jigger. The mostly used combination for all these purposes is Naphthol AS-SW and Fast Red KB Base, which produces on all the above mentioned cotton materials brilliant and fast-to-caustic boiling red shades. Because of its high substantivity the AS-SW can be used for the production of shades which possess a most superior fastness to crocking, since such naphtholated goods can be submitted to a rinsing with an alkaline salt solution which removes the excess of the naphthol liquor not fixed on the fiber, without impairing the depth of the shade. It stands to reason that these qualities go hand in hand with a highly increased fastness to crocking. With the lesser substantive naphthols this can only be attained either by drying between the operations or by a heavy mechanical soaping after the developing which, of course, is impossible in working on dyeing machines. I personally believe that with the introduction of the Naphthol AS-SW the long discussed problem of fastness to crocking has been brought to a happy solution.

At the end of my discussion about the dyeing with naphthols I want to mention the considerable simplification which has been accomplished by the introduction of the so-called "fast color salts" which are ready diazotized bases in stable powder form. Of the sixteen bases which Griesheim produces we have now succeeded in bringing on the market ten of the most important ones in salt form. The method of application is the simplest imaginable; one has only to dissolve the fast color salts in water and pass the naphtholated goods through the solution. One eliminates, therefore, the tedious process of diazotization with its many chemicals such as nitrite, acid, ice, neutralizing agents and so on, as well as losses of material by incomplete diazotization, and one assures accurate and faultless working, which is of particular value where unskilled labor has to be depended upon. The dry diazo salts are practically stable for years and their solutions, containing a stabilizer, keep better than a normally diazotized base.

Calico Printing with Naphthols

A second field for the application of naphthols, equally as important as dyeing, is *calico printing*. Here we have to differ between the discharging of plain dyed goods and the actual direct printing. It is quite natural that with the immense amount of possible shades from the eight naphthols and sixteen bases the dischargeability varies according to the chosen combination. Generally it can be said that most of the shades give a good white discharge; best are the combinations with Naphthol AS-BS, nearly as good, the ones with Naphthol AS and AS-RL. Not dischargeable are the yellows from Naphthol AS-G; just as these are, as previously mentioned, perfectly stable to kier boiling with caustic, they are also not affected by any reducing agents.

The printing of naphthols can be carried out either by direct printing of thickened diazotized bases or fast color salts on naphthol prepare, or in certain cases by printing of thickened naphthol solutions and subsequent passage through a diazo bath.

The Rapid Fast Colors

A third application is, however, the most important and interesting one, namely, the so-called Rapid Fast Colors. These latter are mixtures of the sodium salts of naphthols with the nitrosamines of bases, the two components brought together in a strong alkaline medium so that they do not affect each other.

Under the influence of weak organic acid the nitrosamines are converted into diazo compounds which immediately combine with the naphthols and form the azo dyestuff. The method of application of the Rapid Fast Colors is, therefore, a very simple one. One only has to print the thickened colors on unprepared cloth, to dry and to fix either by aging overnight or by a steaming for two or three minutes, which, of course, allows larger production than the aging.

After this fixation one passes through a bath of weak acetic acid and Glauber salt in order to develop the color, then rinses and soaps. A further simplification of the process can be accomplished by steaming in the presence of acetic acid fumes, which can be produced by placing shallow vessels with the acid into the Mather Platt.

As the process of steaming under pressure in the presence of strong alkali can be compared with a kind of kier boiling and as the Naphthol AS colors are, with exception of the yellow and the red from AS-SW and Red KB Base, somewhat affected by this process, it is advisable to add to the printing paste for counter action a certain amount of sodium chromate. In some cases, chiefly with the Rapid Fast Red GL, Ludigol—a most easily reducible organic compound—acts in the same way. By addition of Ludigol it is even possible to print the Rapid Fast Red next to basic colors which require a steaming of ½ to 1 hour. In the range of the latest Rapid Fast colors we have even developed two representatives—the Rapid Fast Bordeaux and Yellow—which stand steaming and can, therefore, be printed with any dyestuff which requires long steaming.

The range of shades which can be produced is the following: The yellow is represented by Rapid Fast Yellow, lately substituted by Naphthol AS-G and Rapidogen G, as the old product, the mixture of Naphthol AS-G, and the nitrosamine of the base did not possess the desirable stableness, and, therefore, had to be replaced by the two single components. For the production of yellow one has, therefore, to add to the Rapidogen G a corresponding quantity of Naphthol AS-G in order to produce the Rapid Fast Color.

For orange shades we bring Rapid Fast Orange RG and for reds the Rapid Fast Red GZ, a yellowish scarlet; further, the Reds 3GL, GL, B and 2B; for pinks the Rapid Fast Pink LG and LB which are derivatives of Naphthol AS-RL and are, therefore, of excellent fastness to light which equals that of vat pinks. For Bordeaux we possess the Rapid Fast Bordeaux B and for blue the Rapid Fast Blue B. As you see from this assortment, the range of shades is quite a large one, but not complete since greens, browns and violets are missing. To overcome this disadvantage Griesheim has worked out an interesting, new, patented printing process which consists of adding the new Indigosol O or O4B to the Rapid Fast Colors and in developing both products simultaneously. As you undoubtedly are aware, Indigosol is not affected by alkali but is transformed immediately into Indigo by acid oxidizing agents, like nitrite and sulphuric acid. The Rapid Fast Colors require acetic acid for their development, which, however, is not strong enough to split up the Indigosol. On the other side, sulphuric acid will not develop the Rapid Fast Colors because azo colors are not formed in presence of mineral acid. We found, therefore, the happy medium to carry out the development of both in presence of formic acid. The fastness to light of these mixtures is, of course, dependent from the fastness of the two components, and here in any case that of the Rapid Fast Colors is superior to that of Indigo and Bromo-indigo.

The Naphthol-azo and Rapid Fast Colors, with their excellent fastness to chlorine and washing and their very good fastness to light, answer the ever-increasing call for faster shades, which—it seems to me—is just as lively pronounced in the United States as it is on the other side. There is no denying that the use of dyes of inferior quality is decidedly decreasing everywhere and that the superior types replace more and more the poorer ones. The Naphthol-azo and Rapid Fast Colors will surely find in this movement the place they deserve.

The large range of articles in woven, plain dyed and printed goods which can be produced, may be illustrated by some patterns from practical work which I have placed here before you and which I invite you to kindly inspect.

I thank you for your esteemed attention and want to add that I am gladly ready to answer any questions, so far as this is possible for me.

Gentlemen, I thank you.

Discussion

Chairman Grandage—Gentlemen, Dr. Rath has extended an invitation to you to ask questions. We should hustle them through and get as many thoughts as possible on naphthol while he is here. He won't be with us very long and we want to get them.

Mr. Carter—Dr. Rath, do you remember in 1914 when Naphthol AS was first shown on this side, there was also another naphthol—Naphthol NA. At this time, as there was a coming shortage of Indanthrene Black, I experimented with a black suggested by Griesheim Elektron

which was produced in two operations, the yarn being first dyed with Naphthol NA and Metanitraniline (Fast Orange R Base), after which it was dyed again with Naphthol AS and Dianisidine (Fast Blue B Base).

This gave a good black which was not affected by three weeks' outdoor exposure in summer. It stood chemic very well but did not stand a kier boil. That is, it stood the bleach usually given to cotton goods woven in the gray with vat colors.

At this time I also made comparative trials of Red Damask woven with yarn dyed with Naphthol AS and Fast Red G base (Meta-nitro-para-toluidine), against Turkey Red Damask. When I saw this paper on tonight's program I looked up my record of these trials.

The Naphthol AS Red was found to stand six weeks' outdoor exposure during July and August, as well as the Turkey Red, both being practically unaffected.

On putting samples through the regular bleaching process, a kier boil of about nine hours with 4 per cent caustic soda, followed by the usual chemic and sour, the Naphthol AS Red was completely knocked out by the kier boil while the Turkey Red came through the whole process practically unchanged.

Another trial was made in which an ordinary boil-off was given. The samples were then chemicked in the yarn process, being in a tank in which chemic testing 0.45 per cent active chlorine was circulated for three hours; then one hour's wash, antichlored with bisulphite one hour, then washed one hour and taken out. In this case the Naphthol AS Red stood perfectly, but the Turkey Red did not stand. Of course, in circulating, the chemic becomes highly aerated. I think Dr. Rath will be interested to see these trials. (Samples passed to Dr. Rath.)

Dr. Rath—From a historical standpoint these are very interesting to me, and it is very well known that the naphthol colors with the exception of the yellow I mentioned and the new red from SW and Fast Red KB, do not stand kier boiling, not even a slight kier boiling. They are damaged by boiling with soda. Turkey Red does not stand chlorine, particularly if the chlorine is acid.

Mr. Carter—There is another matter in connection with bleaching by circulating the chemic solution in a tank. It is usual to run a week, freshening up the chemic for each lot, the strength being controlled by chemical test. As the chemic gets older its activity increases.

A trial which I made in connection with this will be of interest.

I made two solutions of chemic, each testing 0.5 per cent active chlorine, one solution was freshly made with water and the other with spent chemic liquor, freshened up to 0.5 per cent chlorine. (The spent liquor used was after a week's work.)

Equal weights of Bleach Fast Turkey Red yarn were put in equal volumes of the two chemic solutions and let lie 1½ hours, then taken out, washed, antichlored, washed and dried.

The Turkey Red in the freshly made chemic solution was not touched, but the sample in the solution made from spent chemic liquor showed decided signs of being acted upon.

My opinion was that this was due to the accumulation of chloramines in the spent liquor.

Dr. Rath—By free chlorine, Turkey Red is immediately destroyed; by alkaline chlorine it is not.

The black you mentioned from NA and the orange and Naphthol AS and Dianisidine is a very old process. I remember the first years of our experimenting with this. Of course, it was very difficult to get quite an even black shade because dyeing with Dianisidine was not as widely developed as it is now. Now we bring the Fast Blue B Base and the Dianisidine in the form of a salt which contains in the right proportions all the necessary chemicals, chiefly the neutralizing agent.

Mr. Carter—The black stood light very well. It stood three weeks' exposure to the weather without any change.

Dr. Rath—Dianisidine shades of Naphthol AS are very fast if you give them a treatment with copper.

Chairman Grandage—Are there any more questions before we go on with our next paper?

Mr. Knowland—In reference to present-day printing in this country, the machine printing is being superseded more or less by screen printing. It is doing away with block printing. I notice a lot of these samples are dyed on a machine. In novelty work they are asking for bright shades. Would it be possible in naphthol colors to complete a line to make the varying shades of green?

Dr. Rath—I have passed around a sample book with the shades.

Mr. Knowland—Are they all machine?

Dr. Rath—You can use it for block printing and hand printing, also.

Mr. Knowland—Are they all developed in the same way?

Dr. Rath—Yes. They are steamed in the Mather-Platt or you let them hang overnight.

Mr. Knowland—The screen printers want to do away with steaming if they can immediately wash them. It gives their cloth a much better feel when handled.

Dr. Rath—You can do that. The colors have to be fixed. They must age for a certain time, possibly overnight, and some develop immediately. There are several colors, for instance, the blue, which can be developed immediately after printing. It is merely necessary that they be dry.

Mr. Knowland—The designs on the screen are not being painted; they are photographed.

Dr. Rath—They don't affect more than other alkaline dyestuffs do. Of course, you have certain difficulties with the wool on your printing table.

Mr. Knowland—That is the secret of the screen.

There is another question I should like to ask. I have been asked repeatedly for reds for shirtings on silk where the red is woven against white. Can it be done?

Dr. Rath—Yes. Naphthol AS colors can be dyed on real silk, but you must protect the silk from the action of the alkali in the usual manner.

Dr. Knowland—Will it stand boiling out?

Dr. Rath—Yes, it stands boiling. It is a pigment dyestuff.

Mr. Knowland—It invariably stains the white.

Dr. Rath—It will stain the white not a red shade, but it will stain yellow.

Mr. Knowland—Yes, it stains yellow—but how do you overcome this?

Dr. Rath—The diazo solution has not been washed out properly. Yellow stain means an excess of Diazo left in the fiber and decomposing after.

Dr. Knowland—Due to insufficient rinsing before weaving?

Dr. Rath—Yes. It requires very good rinsing.

Mr. Knowland—I can't tell you exactly what I have in mind, but the color is put on both sides of the fiber. You take a very thin Georgette and print sky blue on one side.

Dr. Flynn—You didn't refer to stencil printing then?

Mr. Knowland—No.

Mr. Brownell—I should like to ask Dr. Rath what would be the difference in the shade if you used AS-SW with a GL base in place of Naphthol AS.

Dr. Rath—SW gives a Bordeaux shade and the AS gives a red shade with the GL base. That is the difference.

Mr. Hales—Is it possible to get with these naphthols as full a shade as Turkey Red on silk?

Dr. Rath—Yes, it is possible.

Mr. Hales—What about bleaching and washing? Do you consider they are as good as alizarine?

Dr. Rath—You know the combination. We give it a number 4 in fastness in the five numbers.

Mr. Hales—In other words, Alizarine Red is still the fast red?

Dr. Rath—Yes, it is still the fastest red.

Mr. Graenicher—May I ask the doctor about fast colors? Is it possible to make them by using some protective colloid?

Dr. Rath—Yes, if you take a protective colloid, as, for instance, glue, then you can protect silk.

Mr. Graenicher—Is it possible to make the colors useful for Celanese?

Dr. Rath—I doubt whether it will be possible to use them for Celanese, because Celanese does not take up naphthol itself. The only way you can dye Celanese is to reverse the process. First, take the hydrochloride of the base and then dye the Celanese fiber with it. It dyes on the fiber and develops in naphthol. You can't dye in the ordinary way with naphthol. I doubt if it will be possible to print Celanese.

Mr. Schaumann—Are these naphthol colors fast to acid cross dyeing?

Dr. Rath—Yes, they are fast to cross dyeing.

Mr. Herrmann—Dr. Rath, one of the requirements which is probably the dyer's chief worry is to match shades in infinite varieties. In fact, the dyer doesn't always know in the morning what shade he may have to produce in the afternoon. As I look through your collection I find a lot of pure spectral colors, but very few of these mauve shades—grays, brown, tan, flesh, copper and the infinite variety of shades which one really sees on the market. What opportunity is there at the present time of reproducing those things economically and with a degree of assurance that the dyer will get his production out?

Dr. Rath—It seems to be that is a drawback in the naphthol series. You can just produce this shade or this combination. There is no possibility by adding some other component to vary the shade. Of course you can, from ASG and a yellow base, by mixing with AS, produce the whole range from yellow to red, through all the oranges, but you cannot have such tan shades and gray shades and so on. You can't do it. You are limited to standard shades. You must produce the shades as they come and not as your customer wants them.

Mr. Grandage—If there are no further questions, we are fortunate in having Dr. Oscar Schmidt with us, and we will ask him to present his paper, "Comparative Dyestuff and Dyeing Conditions Here and in Europe."

. . . Dr. Schmidt's paper proved to be a burlesque, which, after some initial uncertainty on the part of the audience as to whether it was intended to be serious, was received with much laughter and applause. . . .

Chairman Grandage—I am sure we have all appreciated Dr. Rath's very able paper and Dr. Schmidt's amusing one. I can't entertain any questions on Dr. Schmidt's paper as he has too many "trade secrets" in it.

I promise you as a member of the Outing Committee that you will hear from us very soon, and we hope that we will have as successful a time as we had two years ago.

Before we close up the year, it occurred to me that it might be well to just say a word or two about the last year's work and how much work it has meant to the Sectional Committee and the officers of the Section to prepare that work for you. The hardest work that has been done during the year has been to secure subjects that most of our members would be interested in. A

number of times we have had papers presented that were of very vital interest to a number of our members and of little or no interest to others. I propose during the summer, with the permission of my aides, the Secretary and the Vice-Chairman, and the rest of them, to submit a questionnaire to the Section and I hope all the members that are able to will fill them out to the best of their ability, and at least send them back with some comments on the subjects that are going to be of most interest to you.

Is there any further business to bring up before we pass a vote of thanks to our speakers?

. . . Upon motion made, seconded and carried, a rising vote of thanks was given to both speakers of the evening. . . .

. . . The meeting adjourned at 10.30 P. M.

D. P. KNOWLAND, Secretary.

APRIL MEETING OF THE RHODE ISLAND SECTION

The April meeting of the Rhode Island Section of the American Association of Textile Chemists and Colorists was held in the rooms of the Providence Engineering Society, 44 Washington Street, Providence, R. I., Friday evening, April 17, 1925.

The meeting was called to order at 8.15 P. M. by Chairman Williams.

As there was no business to be transacted at this meeting, the speakers were immediately introduced.

A. R. White, 2nd, manager of the Mount Hope Finishing Company, gave a most interesting and instructive talk on the "Advancement in Research Work in Cotton Textiles in England.".

Immediately following Mr. White's address, Dr. Baker, chief chemist of Wallace & Tiernan Company, spoke on the subject of "Chlorine and Chlorine Compounds in the Bleaching of Textiles." Mr. Baker's address, published below, was of great interest, as he dealt to some considerable extent in the chemistry and history of chlorine and its uses.

Appreciation of the speakers was shown by a rising vote of thanks.

The meeting was then adjourned.

Respectfully submitted,

ALBRO N. DANA, Secretary.

The Bleaching of Textiles with Chlorine and Its Compounds

By DR. J. C. BAKER

Wallace & Tiernan Company, Inc.

The bleaching of textiles is one of the old industrial arts, and the use of chlorine for this purpose has been practised since the time of Bertollet, shortly after the American Revolution. Bertollet found that by bubbling chlorine through a solution of soda ash until carbon dioxide began to come off that he obtained a very potent bleaching solution. In fact, he had produced sodium hypochlorite in a form very similar to that which is used to-day, and the improvements in the process which have been made since this time have been chiefly modifications and variations of the cooking, the bleaching technique and improvements in the mechanical method of handling the reagents and textiles.

In the bleaching of cotton there are numerous things desired. In addition to a perfect white color the cloth must be so treated that it will not revert in color with age; that it will have a soft feel capable of handling in other processes; that its strength be not impaired; that the waxy coloring of the cotton fiber be removed and that the motes, if not entirely removed, so treated that they do not injure the appearance of the cloth.

PIGMENTS PRESENT IN COTTON FIBER

To accomplish all these things in a simple and reliable process is not an easy matter. The coloring matter in the cotton fiber is due to the natural vegetable pigments which are found in many forms of living substance. Similar colors are found in many food products, and the bleaching of these same colors is accomplished in many other industries. They are essentially in nature carotin, this name covering a great group of substances. The cotton plant has in the blossom a characteristic yellow color which is present as gossyptin—a glucoside soluble in hot alcohol. There is also present in the cotton the natural pigment of the seed which is found in cotton-seed oil. This pigment is precipitated by alkalies in the purification of cotton-seed oil and doubtless precipitates on the cotton fiber in the alkaline cook.

There is also present zanthopyl from chlorophyl degradation, this pigment having a brownish color. There may also be present in the frosted cotton chlorophyl, giving a bluish or greenish tinge to the cotton. These pigments are most of them soluble in organic solvents and are characterized by solubility in cold or hot alcohol, solubility in fat, and insolubility in water. They may be associated with the waxy coloring of the cotton or may be associated with the fiber itself. These colors all undergo change while the cotton is on the plant in the field, and often while in the bale there is a slow change, the color tending to become dull and more difficult to treat.

If the bleaching of cotton were concerned only with the removal of these coloring materials it would be a fairly simple matter as they all respond readily to bleaching reagents, but the presence of the mote in cotton complicates the bleaching problem and necessitates cooking to pulp this material and render it bleachable. Also the

waxy material complicates the problem and its saponification is very difficult and necessitates special treatment in the cook.

Many variations in the cook are in use, depending upon the result desired. The most common method is the caustic soda cook in which is present some soda ash and soap. Other methods require a lime cook followed by acid and then soda.

One point of great importance in all cooking processes is the elimination of all oxygen, either gaseous or dissolved in the cooking liquor. The cooking liquor should be handled in such a manner that no dissolved oxygen is present when introduced into the vat, thereby avoiding any tendering of the goods. The cooking process, in addition to removing the waxy coloring and softening the mote, dissolves and saponifies some of the coloring matter so that the subsequent bleaching process becomes much simpler.

The bleaching of textiles may be accomplished by calcium hypochlorite made either by dissolving bleaching powder or by dissolving chlorine in milk of lime. There is a slight difference in the action of these two reagents, bleaching powder being somewhat more alkaline and slower in its action. Both methods give somewhat similar results and are mainly used in recirculation methods where raw cotton is being treated.

Use of Sodium Hypochlorite

The most common reagent used in the bleaching of textiles is some form of sodium hypochlorite. This compound in its simplest form is made by dissolving chlorine in caustic soda. If pure reagents are used and the reaction carefully adjusted, practically pure sodium hypochlorite is obtained. This solution has very little buffer action but can be used with great success in bleaching if properly handled. However, if there is much alkali in the goods going to the bleaching vat, its effect will be noticed in a slowing of the bleaching action. Also with this reagent the effect of carbon dioxide is very great as a small amount absorbed from the atmosphere or generated in the cloth will greatly accelerate the reaction. A solution of sodium hypochlorite is a very interesting chemical workshop. Sodium hypochlorite hydrolizes to give a small amount of hypochlorous acid. The ionization of hypochlorous acid being less than that of water, hydroxyl ions are released. If to a solution of sodium hypochlorite one adds a very small amount of acid so that a small amount of hypochlorous acid is released and then applies this solution to a substance to be bleached, the hypochlorous acid acts with great speed, but does not disappear from the solution because the amount of hypochlorous acid is continually renewed by the strong hydrochloric acid which is formed from the bleaching reaction, combining with the sodium and releasing more hypochlorous acid. Such a solution is a source of a nearly constant concentration of hypochlorous acid throughout the bleaching period, the hypochlorite being the reservoir from which the acid is drawn.

However, if small amounts of acid or alkali are continually introduced into this solution during the bleaching, then this action is altered and the supply of hypochlorous acid either increases or decreases. To avoid this difficulty most bleachers use in the solution some buffer material which may be either carbonate, borate or phosphate or any other salt of a weak acid which by its presence can absorb the addition of extraneous amounts of acid or alkali. The most commonly used buffer is soda ash. In the ordinary method of making bleach the equivalent amount of caustic acid used is less than the amount of chlorine so that some of the chlorine reacts upon the soda ash and forms some sodium bicarbonate. The final result of making a solution of bleach from caustic and soda ash is a mixture of sodium hypochlorite, soda ash and sodium bicarbonate with a very minute amount of hypochlorous acid present.

Because of this buffer the amount of hypochlorous acid present during the bleaching action does not vary and its action is much better controlled. A similar solution can be obtained with probably more accuracy by making up a pure sodium hypochlorite from chlorine and caustic, and then adding to this solution as used a mixture of soda ash and sodium bicarbonate so that the desired final reaction of the solution is obtained. By this method considerable economy of alkali material and sometimes chlorine is shown, this being because a more accurate adjustment of the solution is obtained and there is no necessity of using excessive amounts of soda ash to take care of variations in the manufacturing process. A further advantage of this method is found because the operator can make a large amount of a stable sodium hypochlorite and can activate it as used by adding the buffer solution.

Stability of Hypochlorite

The subject of stability of sodium hypochlorite is an interesting one. It is found that a pure solution of sodium hypochlorite containing a very slight amount of excess caustic alkali is a very stable solution. If the materials used in manufacture are sufficiently pure and the conditions adjusted properly, a 3 per cent solution of sodium hypochlorite can be kept for a year or more with very little loss. If excessive amounts of alkali are added to this solution, its stability is not so great as the tendency to give off oxygen is increased. Soda ash buffer is often used to offset the absorption of alkali by the container.

However, there are on the market now solutions of

sodium hypochlorite containing as much as 14 per cent available chlorine which have considerable excess of alkali which will lose only about 10 per cent of their chlorine per month under ordinary conditions. It is of no advantage to the practical bleacher to make such strong solutions as considerable difficulty is encountered in manufacture. Large amounts of heat are generated and ice must be used to cool the solution during manufacture. The strongest solution practical to make without the use of ice is approximately a 3 per cent concentration, and most mills work with solutions containing 3 per cent or under of available chlorine. The purity of reagents used in making sodium hypochlorite has a great effect upon its stability. The sludge which comes from ordinary caustic and also from most water supplies by precipitation of calcium or magnesium and iron by the alkali causes the solution to decompose. Also the organic matter present in the reagents or in the water absorbs chlorine. Very frequently a solution freshly made will lose a considerable percentage of its chlorine overnight, after which little further loss will occur. This is due to the absorption of chlorine by the organic matter in the water or alkalies.

The Flash Test

In the preparation of bleaching solutions, they are often controlled by specifying the amounts of available chlorine and alkali. There is, however, a very simple method available which enables one to judge not only the rapidity of bleaching but also the stability of the solution This is commonly called the phenolphthalein flash or the flash test. If alcoholic solution containing 1 per cent of phenolphthalein is dropped into some of the sodium hypochlorite it will turn pink after which the bleaching action of the hypochlorite will destroy the phenolphthalein and the pink color will disappear. The rate at which the pink color disappears when chlorine concentration, temperature and all other conditions are constant is a function of the hydrogen-ion concentration of the solution and is a direct measure of its speed of bleaching. If too much alkali is present the pink color takes a long period of time to fade and the solution will be very unsatisfactory because of bleaching too slowly. If no pink color develops, the solution is too acid, will decompose very rapidly and probably bleach faster than is desired. Usually a very short flash of a few seconds is used for controlling bleaching solutions. In order to make the test quantitative the temperature of the solution, the amount of solution used and the amount of phenolphthalein solution must always be kept constant.

There are many other chlorine compounds which can be used in bleaching textiles with more or less success. Chlorine gas itself has been used by exposing damp cloth to the gas in a closed chamber. In case a certain amount of alkali is left in the cloth the reaction is more satisfactory. This, however, is not a commercial method.

Bleaching with Hypochlorous Acid

A method that is being used in the bleaching of rag stock and paper pulp with considerable success is a solution of hypochlorous acid. This is made by dissolving chlorine in water to about a 3 per cent solution and then passing the chlorine water through a tower containing limestone. Carbon dioxide is released and passes off at the top of the tower. The solution of hypochlorous acid formed is very stable as long as it is kept in contact with limestone. It has great bleaching velocity, and with textiles which have been properly cooked is capable of giving satisfactory results. However, the use of this material in the bleaching of textiles has not been entirely perfected.

Advantages of this method of bleaching are the great simplicity of operation, the speed of bleaching and economy of reagents as limestone is very much cheaper than soda and less chlorine is required. The disadvantages are that some calcium is present which needs to be washed out after bleaching. The effect on the motes is not entirely satisfactory unless the cook has been conducted in a manner that will make the mote subject to treatment by this method. Lastly the solution is dilute and a larger volume of bleaching liquor must pass into the bleaching vat than is customary so that to carry away this volume of bleaching liquor, the cloth going out of the vat must have more moisture than the cloth entering the vat.

A great many other forms of hypochlorite could be made and have been used, such as potassium, magnesium, barium, and strontium, but none of these have met with commercial success.

Applicants for Membership

Active

Brandt, C. D., assistant superintendent and chemist, Lowell Bleachery, S. Experiment, Ga. Sponsors: J. B. Smith and P. F. O'Neill.

Logan, George H., superintendent, Swiss Hosiery & Dyeing Company, Louisville, Ky. Sponsors: A. R. Thompson, Jr., and W. H. Willard.

Mueller, C., assistant superintendent, dye and bleaching, Bibb Manufacturing Company, Macon, Ga. Sponsors: A. J. Jorgensen and C. H. Stone.

Supple, William H., dyer, Mateawan Manufacturing Company, Beacon, N. Y. Sponsors: D. P. Knowland and J. J. Sokolonski.

Changes of Address

Baldwin, Harold W., 110 Crest Avenue, Revere, Mass.

Hellmuth, Adam A., 1239 Boulevard East, West New York, N. J.

Del Plaine, P., 363 Randolph Street, Holbrook, Mass.
Hood, Leslie N., care of Union Bleachery, Box 598, Greenville, S. C.
Riley, Jr., B., P. O. Box 1474, Providence, R. I.
Richardson, Jas. M., 75 Pleasant St., Manchester, N. H.
Stubbs, Samuel A., 30 Oak Park, Claremont, N. H.
Triesnor, Robert C., 646 Eagle Avenue, Bronx, New York City.
Trimble, Warren W., Garnerville, N. Y.

CORRECTION

In connection with the paper appearing in the Proceedings of the April meeting of the Southern Section in the June 1st issue, entitled "The Dyeing of Artificial Silk," by E. F. James, the name "Textile Dye Works" appeared beneath the author's name. This should have read "Textile Silk Dye Works."

ASSOCIATION BADGES

The lapel button badge illustrated herewith is worn by members of the American Association of Textile Chemists and Colorists.

The three "rings" are enameled in red, yellow and blue, respectively, and the initials and border are brought out in gold. The general effect is exceedingly attractive.

Badges may be secured at $1.50 each by addressing the Secretary.

WALTER E. HADLEY,
5 Mountain Avenue,
Maplewood, N. J.

AMERICAN
DYESTUFF REPORTER

Devoted to the use and application of dyestuffs and the mechanical equipment incidental thereto.

LOUIS A. OLNEY
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GRAFT

HIGH above its far-famed prestige as a paragon of wealth and strength, American business cherishes its equally universal reputation for integrity, the natural offspring of a basically sound character. But despite the activities of the Federal Trade Commission and a host of enthusiastic business associations sponsoring golden ideals of trade and commerce, we are reminded at times that a distinctly dishonest element still insidiously plies its way in business. It threatens to destroy the very basis of all buying and selling—confidence. It boldly withstands the angry attacks of the business idealists. It defies organized attempts to wipe it out, and it often seems discouragingly powerful. A notorious factor in this element is the commercial grafter.

Graft in the dyestuff and textile trade has been the subject of veritable volumes of editorial diatribes. But the very persistence of the practice excuses equally persistent censure.

In recent years both those who bribe and those who accept bribes in the mill have met their enemy in the technical mill man—a species whose members are not born at the rate of one per minute. At bottom the supression of the graft evil in the mill directly depends upon a close supervision over the purchase of all supplies by a man with sufficient technical knowledge to literally "scare" his dyers and finishers into honest dealing. In the larger mills the routine testing of dyes and chemicals tends to assure honest buying and to promote an independent attitude toward any employee who may injudiciously fall under suspicion. Here the practice is rare.

But where graft does exist, we feel that very often advantage is not taken of every opportunity to squelch it. There is a passive conniving at the evil which is not by any means the best policy. Mill superintendents who evade their responsibility in this respect should be urged to a more stringent attitude by the thought that graft often implies the purchase of inferior products at high prices or the wanton waste of supplies. It plainly represents, therefore, a steady leak in profits which in several items might total a not inconsiderable sum at the end of a year.

But aside from any financial loss to the mill there is the encouragement of the evil to the detriment of the industry in general. It is only by supressing each individual case that we can succeed in reducing the practice to the minimum in the industry as a whole. The heads of the mills help to encourage or suppress graft by their attitude toward the practice. Those who "stand for it" are increasing the difficulties of those who courageously oppose it.

WITHIN THE LAW, BUT ODOROUS

A RECENT editorial commended one of our large dyestuff manufacturers for taking steps against patent infringement by the illegal importation and sale of dyestuffs protected here by American patent rights. The dyestuff "bootlegger" who thus infringes on patent rights must face the law. The owner or licensee of the infringed patents can bring legal force to his aid in combating the practice. It is plainly a matter of catching the offender with sufficient evidence to institute proceedings against him for an order to cease and desist.

There is another, almost identical practice, also termed "bootlegging," which, however, remains beyond the law's reach. The offender in this case pays the royalty on his importations and thus incurs no legal punishment. But he does violate business ethics in that his competition in the dye trade is unfair. He, like the illegal "bootlegger," purchases his dyes abroad at prices far below their actual market value, generally from reparations sources, brings them into this country and offers them in the market at prices five or ten per cent lower than the legitimate distributor of the same colors. Because of this low purchase price he is able to pay duty and royalty, in case his dyes are patented here, and still undersell American dyestuff manufacturers and legitimate importers. One dyestuff man inelegantly but expressively described it as "business that smells."

Those who indulge in such bootlegging are not urged by any intent to injure the American dyestuff industry. To them it is business, pure and simple. They are merely using an opportunity to add a dollar or two to their incomes. Because this dollar may often be multiplied by thousands does not to them make their business any more reprehensible. In this they are right—ethically, it doesn't. But the American industry suffers financially in proportion to the amount of trade which the "bootlegger" takes from it.

The resultant competition affects American manufacturers and legitimate importers alike, not so much because of its volume as because of its tendency to make consumers dissatisfied with current prices. The industry in general can never be in a perfectly healthy state while the evil exists.

The fault in most cases lies with those who purchase the "bootleg" colors. Like the graft evil, power to stop it lies with the "ultimate consumer."

LOWELL TEXTILE SCHOOL GRADUATION

Thirty-one students comprising the class of 1925 graduated from Lowell Textile School on June 9th. Graduation exercises were held in Southwick Hall of the school and were attended by one of the largest audiences that have been present at the annual commencement of the school.

Charles H. Eames, president of the school, made the opening address, interestingly reviewing the growth of this country's textile industry and emphasizing the increasing importance of Lowell Textile School and similar institutions in technical education. Following President Eames' address the audience listened to a very interesting talk on economic condition by Clifford H. Anderson, president of the Associated Industries of Massachusetts.

Medal and Awards

A gold medal was awarded by the National Association of Cotton Manufacturers for proficiency in the Cotton Course, and the regular prizes were presented by the Saco-Lowell Shops, Edward A. Bigelow and Herbert Currier for theses and high scholarships.

The Textile Colorist prize of $100 for the best thesis on a textile chemical subject was awarded to Frederick W. Hibbard, of Lawrence, and Robert E. Sargent, Haverhill.

The annual Louis A. Olney Book Prizes, awarded for theses and highest scholarship to students of the Textile Chemistry and Textile Coloring Course were presented as follows: Senior class prize to Haven A. Morrison, Lowell; senior honorable mention, Parker H. DelPlaine, Lowell, and George W. Pierce, Somerville; junior class prize, first to Stephen K. Ford, Bradford, and second to Chester W. Meyers, Billerica, with honorable mention to Thomas J. Tarpey, Somerville, and Clarence Hooper, Shirley; freshman prizes, first to John V. Killheffer, North Caldwell, N. J., and second to Robert Burns, Easthampton, with honorable mention to George Chester Ward, Andover, and John M. Way, Concord.

Theses

The theses prepared by students of the Chemistry and Textile Coloring Course covered the following subjects:

Maurice Sidney Baker, Dorchester, "Determination of an analytical method for the separation of tartrazine from the fast light yellows."

Joseph Bailey Crowe, Lowell, "Development of an improved method for the determination of organically combined SO_3 in sulphonated oils."

Raphael Cohen, Lowell, "The Action of Ultra-Violet Light on Wool."

Parker Haywood DelPlaine, Lowell, "Study of artificial silks, especially with references to their dyeing qualities." (With George W. Pierce.)

Frederick William Hibbard, Lawrence, "A study of certain derivatives of paracymene, with special reference to possibilities of utilization." (With Robert E. Sargent.)

Haven Asa Morrison, Lowell, "General consideration of the viscosity of starch solutions."

George Whitwell Pierce, Somerville, thesis with Parker H. DelPlaine.

Harold Nelson Runnels, Concord, N. H., "The fastness to light of goods carbonized after dyeing."

Robert Edward Sargent, Haverhill, thesis with Frederick W. Hibbard.

Lui Jorge Villa, South America, "A study of the dyeing properties of nitrated and denitrated cotton."

Graduates of the Chemistry and Dyeing Department, Lowell Textile School. Left to right—R. E. Sargent, J. B. Crowe, H. A. Morrison, M. S. Baker, L. J. Villa, G. W. Pierce, F. W. Hibbard, H. N. Runnells, P. H. DelPlaine, and R. E. Cohen.

Highs and Lows in a Busy Chemist's Life Part VI

IN color matching in dyehouses there is one thing that I have learned that is worth passing on, for it seems to be very little known. The wearing of an ordinary eyeshade is of astonishing assistance in matching colors. It keeps troublesome light from the eyes and permits better concentration on the sample. Of course, it will not prevent a red brick wall's reflecting a confusing light where it isn't wanted, but it will very often cut off from the eye an undesirable reflection coming from snow, a water surface, or a white building. Since such reflections often affect the eye more than they do the sample it becomes a real aid to matching. It is difficult to emphasize how truly surprising the result may sometimes be, enabling matches to be made which would otherwise be extremely difficult.

One day we had a very striking illustration of the difference between per cent on the goods and per cent in the bath, and at the same time an object lesson on care in the use of wartime materials.

A union black worked very well on a cotton and wool twist yarn when dyed in a Klauder-Weldon machine, but when the same color was used on similar yarn packed in a vacuum machine, the whole lot came out tender.

Alkalies tender wool, so the problem resolved itself into a quest for alkali. All of the dye assistants were O. K., and therefore it became a question of the dye itself. Indicators would not work with the dye solution, but salting out the color and then testing, showed a strong alkali reaction, due to soda ash present in rather large quantities, apparently as a restraining agent.

In the open machine with its longer bath, the concentration was not high enough to do any harm. But in the closed machine, with a heavy formula and less liquor there was a sufficient alkali concentration together with favorable time and temperature factors, to attack the wool and seriously tender it.

Luckily we were not responsible, and so we learned again at somebody else's expense that dyes may have to have specific tests as to their uses for certain purposes, as well as the routine tests for strength and shade.

Soaps were always a mystery. It was very easy to tell how much alkali and fat there was, but when it came to predicting how the finished scoured goods might smell a year later, we were not so sure of ourselves.

We did succeed in getting rid of the one that had only 20 per cent fat and finally of the fulling soap that was below 40 per cent, putting in its place a standard make that tested around 60 per cent and figured nearly fifteen dollars less net cost per barrel. Not a bad item to our credit when using carlots.

The mysteries of soaps were, if anything, surpassed by the mysteries of their salesmen. Reiterated claims that a soap was a pure palm oil soap were finally broken down to this, that the soap was a mixture of fats, but the palm oil used was pure.

Or the other one who backed me into a corner and was ready to spend most of the morning arguing that his 88 per cent soap really had a per cent more than the other fellow's, so we could use less of it. Just as though anybody in a mill would bother with the difference when using a few pounds of soap once in a while.

Testing dyes is a business about which we can always learn and always argue. I was once constrained to do a little unusual thinking, through hearing of some individual who insisted that five grams was the proper amount to weight out for a stock test solution, it being considered more correct than the usual one or two grams.

Why, thought I, should one need five grams of dye to properly test a can or keg, when only one gram of coal is the standard amount in the final test to represent a carload, or even five or ten cars? The final answer will be that the coal sample was really several hundred pounds and that this has all been carefully mixed, and ground and mixed, time and again, until the final pulverized sample is reduced to the bulk of a four-ounce bottle, so that each one gram that is weighed does really represent every part of the shipment.

But dyes, too, are sampled in four-ounce bottles, or still smaller containers, and anyone can weigh out one gram of dye from a bottle as accurately as one gram of coal. The one gram sample is simpler, too, because dyes vary in solubility, and with the less soluble it avoids solution difficulties or unwieldly volumes, tending to a desirable uniformity of procedure.

These thoughts with the facts that I have actually found chunks of salt and chunks of unground dye weighing a goodly fraction of a pound in dye deliveries, suggests that more thought should perhaps be taken in sampling dyestuff packages. We may not always have just the same purpose in mind, and we should always remember that nearly every dye is a mixture, at least with some inert material for standardizing. In shipping, there may be a tendency for the heavier and lighter portions to segregate in different parts of the package, creating local areas (or volumes) of different strengths.

If the purpose of a dye test is to determine the total color strength in the container, it should certainly be sampled in various parts of the package, the amounts taken being made into a thorough mixture.

If, on the other hand, the tester works on the theory that dyes are supposed to be uniform and that a sample taken anywhere in the package should represent the lot, may he not, perhaps, fool himself, or be unfair to the seller? For extreme requirements of uniformity, perhaps the dye should be put through a mixing barrel in the mill, as insurance against original incomplete mixing, or change during transit. Any small sample then should certainly represent the lot.

But, when all is said and done, the errors (not mistakes) in dye testing are not in weighing and measuring, but in judging the final results, and in colloidal factors about which we still know mighty little.

(Part VII will appear in an early issue)

Dyestuff Tables

A List of the Principal American-Made Colors with Their Properties and Fastness Characteristics

[NOTE: The publishers of The REPORTER have secured more or less complete data in regard to many of the dyestuffs which are at the present time being manufactured in the United States. As the following material has been brought together from various sources, they realize that errors are quite likely to appear, and it is earnestly requested that any readers who find errors or who are able to give additional information will communicate with the publishers in order that this material may be made as correct, complete and up to date as possible. These tables will appear serially until all colors upon which we have data shall have been covered. The publication of this data was begun in the issue of March 10, 1924.]

ALPHAZURINE FG

(Schultz No. 506)

COMPOSITION: Triphenylmethane.

SPECIALLY SUITABLE FOR: Wool and silk as a level dyeing shading color.

USUAL METHOD OF DYEING: With Glauber salt and sulphuric.

SHADE: Very pure clear blue; less greenish than S-543.

SHADE BY GASLIGHT: Greener.

SOLUBILITY: Excellent.

LEVEL: Excellent.

EXHAUSTS: Exhausts more quickly and better than the Patent Blues.

FASTNESS TO:

Acid: Moderate; dilute sulphuric turns it yellowish green.
Alkali: Fast.
Carbonizing: Turns greener.
Cross-Dyeing: Moderate.
Fulling: Unsatisfactory; better when top-chromed.
Ironing: Inferior to Patent Blues.
Light: Moderate; about the same as Patent Blue.
Potting: Moderate.
Rubbing: Fast.
Steaming: Fast.
Sulphur: Slightly redder.
Washing: Moderate; better when top-chromed.
Water: Fair.

DYED BY OTHER METHODS: Can be used for shading top-chrome colors.

ON UNIONS: Gloria: The wool is dyed greener than the silk. Cotton-wool unions: Dyed acid, the cotton is tinted slightly.

ON OTHER MATERIALS: On silk: Dyes an intense blue in a sulphuric acid bath; the shade is not fast to water or washing. Lakes: Precipitated with barium chloride, suitable for wall paper; also lithograph and letter-press colors.

PRINTING: Suitable for wool and wool-silk printing (sour). No interest for discharge printing, as the discharged places stain again on exposure to the air. Tin salts have no reaction on the color, therefore the dye can be used for color discharges.

COMPETING PRODUCTS: Made in the United States by National Aniline & Chemical Company, under name of Alphazurine FG.

MAGENTA

(Schultz No. 512)

COMPOSITION: Triphenylmethane.

SPECIALLY SUITABLE FOR: Wool, especially flannel and knitting yarns.

USUAL METHOD OF DYEING: Cotton. On wool: With acetic acid at 190 deg. On cotton: On tannin mordant.

SHADE: Varies with the purity of the color.

SHADE BY GASLIGHT: Yellower.

SOLUBILITY: Improved by acetic acid.

FASTNESS TO (on Cotton):

Acid: Fast to acetic acid only.
Alkali: Slight.
Chlorine: Destroyed.
Ironing: Changes shade slightly.
Light: Moderate.
Rubbing: Crocks.
Washing: Fairly fast for color of this class.

FASTNESS TO (on Wool):

Fulling: Moderate.
Light: Moderate.
Perspiration: Fast.
Rubbing: Crocks.

OTHER PROPERTIES: Used for topping substantives on cotton.

ON OTHER MATERIALS: Silk: Dyed with boiled-off liquor broken with acetic. Artificial silk: Dyed with acetic acid. Paper: Suitable for all kinds of pulp, sized or unsized; gives cheap, heavy shades with a yellowish cast. Jute: Dyed neutral at 140 deg. Fahr.

PRINTING: Suitable for calico.

DISCHARGING: On cotton: White with chlorate discharge; white with glucose soda-lye discharge. Gives color discharge with tin.

COMPETING PRODUCTS: Made in the United States by Albert David Chemical Company, under name of Fuchsine Crystals; Sherwin-Williams Company, under name of Fuchsine Crystals; E. I. du Pont de Nemours & Co., under name of Fuchsine Powder; Calco Chemical Company, under name of Fuchsine RS Crystals.

METHYL VIOLET

(Schultz No. 515)

COMPOSITION: Triphenylmethane.

SPECIALLY SUITABLE FOR: Cotton and wool.

USUAL METHOD OF DYEING: On cotton, with tannin mordant; on wool, neutral or with a little acetic acid.

SHADE: Intense violet.

SHADE BY GASLIGHT: Much redder.

SOLUBILITY: Acetic acid aids solubility.

LEVEL: Level on wool.

FASTNESS TO (on Cotton):
Acid: Fast against acetic only.
Alkali: Slight.
Chlorine: Slight.
Ironing: Fast.
Light: Very moderate.
Rubbing: Crocks badly.
Washing: Fair, topped with tannin.

FASTNESS TO (on Wool):
Acid: Fast.
Alkali: Slight.
Carbonizing: Fast.
Fulling: Slight.
Light: Same as cotton.
Perspiration: Slight.
Rubbing: Crocks slightly.
Steaming: Fast.
Sulphur: Bleeds badly.
Washing: Bleeds badly.

OTHER PROPERTIES: Used for tinting white worsted top to correct shade. Special form used for indelible pencils.

DYED BY OTHER METHODS: Used in some bleachers' blues.

ON OTHER MATERIALS: On silk: Dyed with boiled-off liquor and a little acetic; not fast to light; fairly fast to water; fast to perspiration, hot-pressing and crocking. On paper: Used for cheap violets, tinting whites, etc.

PRINTING: Suitable on cotton and wool.

DISCHARGING: On cotton, chlorate process gives a white discharge. On cotton, glucose soda-lye process also gives white discharge.

COMPETING PRODUCTS: Made in the United States by E. I. du Pont de Nemours & Co., under names of Methyl Violet, Methyl Violet Base, Methyl Violet AA Powder and Methyl Violet NE and NP; Albert David Chemical Company, under names of Methyl Violet 2B and Methyl Violet 5B Conc.; National Aniline & Chemical Company, under names of Methyl Violet 2B Crystals, Methyl Violet 2B Lumps and Methyl Violet 2B Powder; Calco Chemical Company, under names of Methyl Violet 4B and Methyl Violet 4BN; Sherwin-Williams Company, under name of Methyl Violet 5B; N. Y. Color & Chemical Company, under name of Methyl Violet EX. Conc.; Newport Chemical Works, Inc., under name of Methyl Violet R Conc. Powder.

CRYSTAL VIOLET
(Schultz No. 516)

COMPOSITION: Triphenylmethane.

SPECIALLY SUITABLE FOR: Cotton dyeing and printing.

USUAL METHOD OF DYEING: On cotton, with tannin and antimony salts; on wool, neutral or with a little acetic acid.

SHADE: Same shade as Methyl Violet 6B (S-517).

SHADE BY GASLIGHT: Redder.

SOLUBILITY: Use a little acetic.

LEVEL: Dyes level on wool.

FASTNESS TO (on Cotton):
Acid: Slight against sulphuric; good to acetic.
Alkali: Slight.
Chlorine: Destroyed.
Cross-Dyeing: Slight.
Ironing: Fast.
Light: Very moderate.
Perspiration: Fast.
Rubbing: Crocks slightly.
Sulphur: Greener.
Washing: Good, topped with tannin.

ON OTHER MATERIALS: On silk: With boiled-off liquor and acetic acid. On jute: Neutral at 140. On artificial silk: At low temperature with acetic acid.

FASTNESS TO (on Wool):
Acid: Fast.
Alkali: Slight.
Carbonizing: Fast.
Fulling: Fairly good.
Light: Very moderate.
Rubbing: Crocks badly.
Steaming: Fast.
Street Dirt: Fast.
Sulphur: Fair.
Washing: Moderate.

PRINTING: On cotton, with tannin. On silk, with tannin.

DISCHARGING: Discharged on pieces prepared with tannin, the color leaves a pure white. Not adapted for color discharge with hydrosulphite. Oxidizing agents discharge the color white. Tin gives color discharge.

COMPETING PRODUCTS: Made in the United States by E. I. du Pont de Nemours & Co., under names of Crystal Violet Base, Crystal Violet Extra Pure and Crystal Violet Powder.

METHYL VIOLET 6B
(Schultz No. 517)

Same fastness properties as S-516.

COMPETING PRODUCTS: Made in the United States by National Aniline & Chemical Company, under name of Methyl Violet 5B Crystals.

SPIRIT BLUE
(Schultz No. 521)

SPECIALLY SUITABLE FOR: Cotton.

USUAL METHOD OF DYEING: On a tannin mordant.

SHADE: Redder than Victoria Blue S-522.

ON OTHER MATERIALS: On wool: Dyed with a little acetic acid or sulphuric acid; fast to fulling and ironing. On silk: Dyed with boiled-off liquor and sulphuric acid.

PRINTING: On cotton, with acetate of chrome; on silk, with tannin.

(Continued on page 445)

Practical Hints on the Production of Bright Colors on Textile Fabrics

Part XIV

Topping on the Padding Machine—Inconvenience of the Plant—Operation of the Machine—Its Advantages—Process with After-treatment—Operation and Advantages

By RAFFAELE SANSONE

IN some mills, and especially in those conducting soaping operations, cloth printing, or the production of special padded goods, it is often preferred to top fabrics dyed with direct colors on the padding machine. A plant for this purpose is constructed on the principle indicated in Fig. 1.

This shows a strong iron construction (F), sustaining two heavy wooden or iron rubber-covered rollers (I, J) kept under elastic pressure through levers and weights (L) and by a hand-screw (S). Below these rollers, which are made for the larger widths of cloth, is placed a deep wooden trough (T), having a long double series of binding rollers placed close together, and of special size, to keep the material as long as possible in the bath of treatment and to allow a greater dilution.

Before this vat is placed a special guiding and opening arrangement (G), with a wooden board conveniently located for use in entering the material (C) from a wooden shaft kept in special bearings under the action of a brake. At the back of construction F is fitted a delivery mechanism (X), which delivers the cotton fabric in the form of pieces to a low wooden table (E). The machine is provided with a handle for releasing all pressure, and is driven by a fast and loose pulley and spur wheels on the bottom roller.

Inconvenience of Plant

This plant, although working excellently when given constant attention, is more liable than the jigger to cause unlevel dyeing. To avoid the production of weaker portions, some dyers add to the bath in vat T from time to time a measured quantity of strong dyestuff solution. If this addition is not made at the correct time, however, the defect is not overcome or deeper shades may follow. Other dyers perform their topping with baths containing a large excess of the basic coloring matter, which may act favorably in some cases.

Under certain conditions a prolonged processing of the fabric in the bath of treatment tends more to cause unlevel dyeing than if a padding machine is used having a small impregnation trough, where the bath of treatment is continually changed and the material comes in contact with fresh dyestuff from beginning to end. A plant offering advantages for this purpose could be constructed as shown in Fig. 2.

This shows three pressure rollers (R), the central one of which is of iron covered with India rubber, while the other two are of copper-plated iron or similar material. The short end shafts of such rollers turn in elastic bearings placed between springs, on the upper of which elastic pressure is applied by lever (E) and weights (J); the action of these can be stopped at any time by simply raising a hand lever (I) at each side of the machine. The bearings of the three pressing cylinders are at each side of the machine in rectangular holes rounded at the bottom and placed in two strong side plates (Z), joined at a certain distance by transverse bars, of which one is indicated by (O). In these they move up and down with the greatest liberty, but cannot turn or be displaced to the right or left owing to pressing plates at their extremities.

Below the pressing rollers is a shallow copper-plated iron trough (K), the contents of which are quite small. This has a large discharge tap on the bottom, a side tube for the disposal of all excess of bath and an upper tube (L) to introduce this from a higher vat (V) placed on a high iron table (F) having at the rear a small platform to which the operators have easy access through an iron stairs, not seen in the illustration. To another vat, placed at a small height at the rear of vat V, the bath pumped from trough K is brought by the centrifugal pump C and tube T. In this other vat

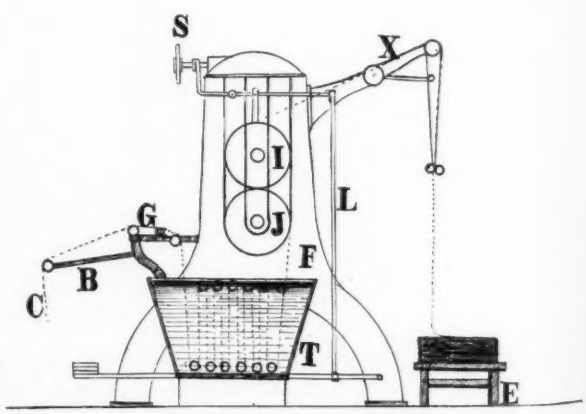


Fig. 1.

the weak bath is strengthened at intervals and added to vat V for immediate use.

Bearings (M) are placed on the front of the machine for entering the material from a wooden beam. Just above these are opening guides (G) and an entering roller. At the back of the machine are arms A, supplied with a movable mechanism P used for transforming the material into pieces.

Operation of the Machine

For working the plant, the operators, after having threaded a short tape through the machine, place on bearings M a wooden beam filled with the first portion of the cloth to be topped that has just been received from the jig, where it has been dyed in the direct color and rinsed; the loose end of this is then joined to the end of the tape just entered.

In the meanwhile, the potting bath is prepared and poured into vat V, the tap of which is opened so that trough K is filled through tube L. When this has taken place and a portion of the bath, by means of pump C, is brought into the vat at the back of V, the plant is started, care being taken to spread out and smooth the cloth as soon as possible.

The cloth then enters trough K, remaining there long enough to turn round the first impregnation roller, being brought at once in the nip of the two lower pressing rollers, and then for a second short contact with the potting bath, after which it is again pressed and passed to plaiter P.

A good portion of the old dye bath collelts little by little in the vat at the back of V, and the operators that have prepared the strengthening solution enter this as soon as a certain reight has been reached in the preparation vat, mixing the bath rapidly and discharging it as soon as necessary into vat V.

Advantages of the Plant

Among the advantages of the above plant can be mentioned the following:

1. The cotton cloth dyed in the direct dyestuff comes in contact with a continually renewed bath of the basic dyestuff and receives the same quantity of this latter throughout the length of the pieces being treated.

2. By carefully arranging the contents of the potting bath and regulating its volume for the treatment of the lot of cloth to be topped, calculating the additions of strong solution to be used for the reinforcing operations, dyestuff waste can be brought down to the minimum.

3. By turning the upper portion of tube T, the potting bath can be delivered into one of a series of collection cisterns in the rear, and the vats of preparation and delivery and rest of the plant can be rapidly cleaned with a little soda lye and some running water.

4. The padding machine can be operated in connection with jigs, treating the rolls of cloth dyed on several of the jigs during the course of a day.

5. By taking the precaution to add the strengthened bath in vat V only when a new piece is being run in, if a slight difference is visible in comparison with the

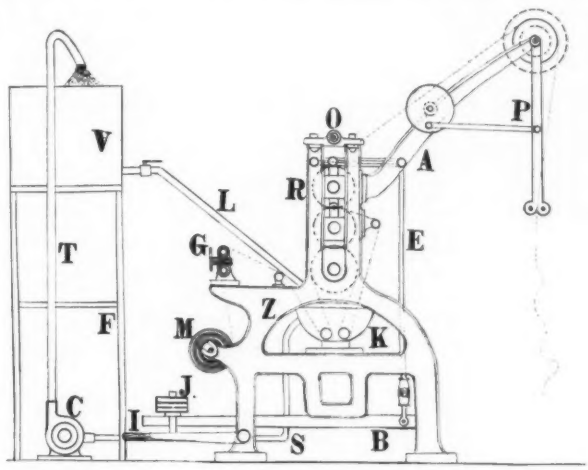


Fig. 2

piece just dyed, this will pass unobserved, or cause little damage.

6. The size of vat V and amount of dye bath prepared and strengthened each time can be regulated so as to be approximately sufficient for two large pieces or four small pieces, thus avoiding the necessity of adding the strengthened bath before any of the pieces treated have been topped.

Process with After-Treatment

In those cases where the process is adopted in which the dyeing operations are followed by an after-tanning, or by a treatment with a second direct dyestuff, it is convenient to conduct the whole dyeing operation in continuous form. For this purpose a plant constructed on the principle shown in Fig. 3 would offer advantages.

This shows five wooden vats (A, B, C, D, E), the first, third and fourth of which are surmounted by three heavy squeezing bowls (F, G, H), the two lower of which are of an iron-silicon metal that resists rust, alkalies and acids, while the central has an iron axis mounted with a thick Indiarubber covering. The end shafts of these bowls turn in ball-bearings lubricated from the side frames and supported on strong steel springs.

Vats A and D are heated with copper-plated iron steam coils and serve for two baths of the direct color, if these are used; or in the case of A for a direct color and in the case of D for after-tanning operations. Vat B serves in this instance for rinsing with water after the dyeing with the substantive colors and diazotizing if necessary; vat C for dyeing with the basic

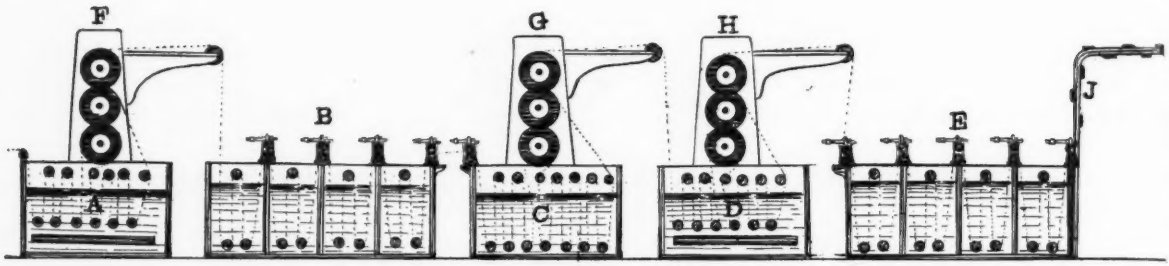


FIG. 3

colors and Vat E for the final rinsing operations with clean running water. This last vat has a simple delivery mechanism (J) that delivers the cloth to a conveyor, on which the material is folded into pieces.

Vats A, C and D have upper and lower guide rollers of glass or similar material, contained in a single bath, while vats B and E are each divided into four compartments with suitable partitions. The first (B) has four light upper squeezing rollers and the second (E) five. For facilitating the passage of the material from the pressure bowls F, G, H to the next bath of treatment, these have before them two projecting arms supporting a guide roller, the position of which can be moved to regulate the tension on the fabric.

Operation of the Plant

For operating the plant described above, after the introduction of a long cotton tape through the different vats, the operators fix to the end of this the beginning of the first piece of the lot of cotton cloth to be dyed and prepare the dye baths that are to serve in each case in vats placed at a certain height, causing these to circulate slowly through vats A, C, D, and all excess to descend into underlying cement cisterns. When this has been accomplished the plant is started.

The fabric is thus brought into the first vat, and when it arrives at the end and begins to mount, the operators lift a lever that reverses the movement to carry it back on the road already made, after which the plant itself assumes a backward and forward movement, continued for a fixed interval and followed by a continuous forward movement for an equal interval of time, the two movements being continued intermittently as long as the plant is worked. By a similar arrangement the material can be kept the necessary time in each dye bath to be dyed the required shade, and in the rinsing compartments to assure a better utilization of the running water.

In vat A it is dyed in the direct coloring matter, being pressed twice between bowls F, and on leaving the same is washed in the four compartments of vat B of all mechanically fixed dyestuff. Vat C conducts a topping with a strong bath of the basic color, leaving a large excess in the material, which is afterward fixed in vat D. The rinsing baths in vat E serve principally for extracting the free tannic acid; they can, however, be employed, if required, for treating with tartar emetic.

Advantages of the Plant

The advantages of the above plant can be indicated as follows:

1. During the process the dyer can observe the cloth being dyed and topped, and can note the different changes taking place, cutting samples near the borders whenever he has any doubt regarding the correctness of the shade produced.

2. As it is possible to fix an excess of the basic dyestuff on the fabric, besides that already taken up by the direct coloring matter, it is possible to obtain brighter shades than with a simple topping.

3. The fastness of the shades obtained is increased by the after-tanning conducted in vat D.

4. The back-and-forth movement given to the material at regulated intervals assures a longer processing in the different baths of treatment, permitting a stronger action of these, which can be employed weaker and exhausted in the case of the direct colors by the use of salt additions.

5. Rinsing vat B can be used when necessary for diazotizing or chromin gthe direct colors dyed, and vat E for fixing with tartar emetic, should this be required.

6. By using bowls composed of an iron-silicon metal, not only do the same cost less than most ordinary bowls at present in use but they last longer.

(To be continued.)

NEWPORT ISSUES FALL SHADE FOLDER

A folder showing the standard shades for fall on worsted material dyed with Newport colors has been issued by the Newport Chemical Works, Inc. Eighteen shades are shown on worsted swatches; on the facing pages are complete dyeing recipes for each shade. The shades comprise Caprice, Lillium, Wild Aster, Saraband, Anatolia, Bokhara, Mosul, Gorevan, Kis Kilim, Serapi, Kara Dagh, Mahal, Dozar, Kabiston, Shirvan, Venetian Blue, Della Robbia and Chinese Blue. This folder is uniform with previously issued inserts for the Newport binder.

Technical Notes from Foreign Sources

Injuring the Woolen Fiber During Carbonization

It is known that when the carbonization process is carried out carelessly, it is a very easy matter to injure the woolen fibers. The wool is said to be "burnt." This means that the chemicals have attacked the fiber and carbonized it, and not that the temperature during the process has been too high, which is most always the case. It is also claimed that the use of magnesium chloride and aluminum chloride for the carbonization of wool has been very small up to the present time, due to the fact that these salts split off hydrochloric acid at a temperature of 120 to 130 deg. Cent. with resulting injury to the wool.

But even when these salts are not employed in the carbonizing process, the danger of injuring the woolen fiber is not entirely removed. The wool may be "burned" as well when it is carbonized with the aid of dilute sulphuric acid, and this is by no means rare. The reason for this is always carelessness or lack of knowledge on the part of the operator.

The carbonizing process itself is considered of two parts, first the preliminary drying and then burning. But considered accurately it involves only one operation, which is the concentration of the dilute sulphuric acid in monohydrate. It is also a fact that the designation of the second part of the carbonizing process as "burning" has led many an operator to believe that he must conduct the process as high temperatures. This is where the difficulty lies.

Thus it must be remembered that the greater the concentration of the sulphuric acid, the slower the process must proceed. This is the natural result of the affinity that exists between sulphuric acid and water. And it is just this slower and smaller removal of water during the drying process that leads many a carbonizer to try to shorten the operation by increasing the temperature. There is nothing which can be said against a gradual rise in the temperature to approximately 40 to 50 deg. Cent., but just as soon as the temperature is raised to the boiling point of water, so as to drive off the last traces of water in the woolen material, then there is great danger that the fiber will be injured. Then again at this temperature the destructive action of the acids on the wool molecule starts and the acid has the effect of removing the water from the wool substance. The result is that a keratin is formed which is a substance that is deficient both in oxygen and hydrogen. The wool is "burnt." This action of burning is produced on the best grade of wool, one that has been most thoroughly washed and that is completely free from suint.

The carbonization of wool that contains suint is an operation that never gives perfectly good results. The acid in this case acts not only on the wool fiber itself, but on the suint and wool fat as well, and in this manner there ensues a reaction which is very complicated and very difficult to control. Just what by-products are formed in this complicated reaction cannot be told with any degree of certainty.

For complete details on this report see Technical Paper Number 20, Supplement to the General Journal for Eiberfeld-Barmen, through Der Textilchemiker und Colorist, Number 9, 1925, page 65.

Waterproofing Sail Cloth

The following method may be employed with good results in the waterproofing of sail cloth. The sail cloth is brought into a bath which contains 1 part of sulphate of alumina and 10 parts of soft water free from iron compounds. The cloth is thoroughly saturated with this solution and the excess squeezed out. Then the sail cloth is impregnated with a solution made in the following manner: Ten parts of soft water, 1 part of soda crystals, and 1 part of light colored pulverized rosin. This mixture is boiled for so long a time that a rosin soap is formed. The rosin soap is salted out with one-third of a part of common salt, the warm rosin soap is skimmed off and is dissolved along with one part of white curd soap made from tallow in 30 parts of water. The impregnated linen cloth is then passed through a water bath for the purpose of removing the uncombined or decomposed soap. The cloth is then dried in the air. (Deutsche Faerber Zeitung, 1925, 541.)

Retting with Urea

At a retting temperature of 27 to 30 deg. Cent. a concentration of 0.25 per cent of urea accelerates the process and eliminates the foul smell as well as larger amounts, and the liquor becomes less acid than in the absence of urea. The product has a better feel and the fibers are better separated than after a normal ret and the waste liquor is an excellent manure.

In another experiment urea was added to the water in a number of large scale rets, and the color feel and yield of the fiber so obtained were very satisfactory. When the flax was difficult to ret the addition of urea caused a marked acceleration. (Faserforsch, volume 4, pages 131-141 and 141-145.)

Dyeing of Acetyl Silk with Insoluble Azo Dyes

Cellulose acetate silk, like natural silk, is dyed with Tartrazine by immersion in a warm liquor which contains the components from which this dyestuff is usually prepared. For example, cellulose acetate silk is dyed to a full yellow shade by immersion for 20 minutes in a liquor at a temperature of 50 deg. Cent. containing 0.75 per cent of sodium dihydroxytartrate and 1.3 per cent of phenylhydrazine together with a mineral acid. An orange shade may be produced similarly by the use of para-nitrophenylhydrazine. Cellulose acetate silk is dyed a

terra cotta shade by immersing it in a liquor which contains acetic acid, meta-tolylenediamine and "nitrosamine." (Journal Society Dyers and Colourists, 1925, 97-8.)

Effect of Temperature on the Dyeing Process

It is a fact that the temperature at which a fabric is dyed has a very potent effect on the character of the color obtained on the same. There are in fact few dyestuffs which give the same results at any definite temperature and variations in the temperature at which the dyeing process is carried out often result in unequal dyeing. This matter of temperature is very important in dyeing fabrics with mixed dyestuffs, and it is essential to test out each individual coloring matter employed in the color bath so as to determine individually what temperature is best suited for them. It has often been found that even the coloring matters that belong to one and the same class of dyestuffs will give different results at any given temperature when used on a certain fiber.

When uneven colorings are obtained with mixed dyestuffs, it is frequently possible to alleviate this condition by reducing the quantity of coloring matter employed. However, this is inadmissible when several pieces of goods are dyed one after the other. In order to obtain the best results it is also always necessary to carry out the dyeing process at the boiling point, but when basic dyestuffs are employed, this practice is entirely inadmissible, for at this temperature this class of coloring matter decomposes with ease. In many cases, on the other hand, the absorption of the dyestuff is enhanced when a small amount of acid is added to the bath containing the basic color.

As far as the cotton colors are concerned the general rule prevails that bright shades are best dyed at moderate temperatures. On the other hand, the average depth shades and the deep colors are dyed at higher temperatures, and the conditions vary in this connection so that some colors are dyed almost at the boiling point of the color bath. But then again, there are many colors which suffer when they are kept for too long a time at the boiling temperature.

There are, furthermore, certain members of the vat class of coloring matters that are very sensitive to temperature changes. When such coloring matters are employed it is necessary to exert the greatest care in maintaining the temperature of the dyeing operation constant and within certain very well defined limits. (Deutsche Faerber Zeitung, 1925, page 540.)

Dyeing with Oxidized Fluorescein

Yellow to red tints are obtained by subjecting fluorescein in the dye bath or on the fiber to direct to indirect oxidation in the presence of metallic salts. The fluorescein may be oxidized by means of chlorates, bromates, or iodates in the presence of reducing agents, such as glucose, saponins, sulphites, and hydrazines, by free bromine or iodine, or by agents such as metallic salts alone or persulphates and metallic salts.

In examples, baths are prepared which contain, in addition to fluorescein, a chlorate, glucose, or sodium bisulphite and acid mercuric sulphate or acetate; an iodate or bromate and sulphuric or hydrochloric acid in conjunction with sodium sulphite and copper sulphate or with aluminum amalgam; free bromine, potassium bromide, and either quinine hydrochloride, pyridinem, quinoline, or gum arabic, and a metallic salt; free iodine, which may be disseminated from an infusion bag in the bath, in acid or alkaline medium with a metallic salt such as a copper salt; corrosive sublimate with or without potassium ferricyanide; acid mercury sulphate; and a persulphate with acid mercury sulphate or a copper salt. Liquid or solid mixtures which contain the above ingredients may be prepared for subsequent solution to form dye baths.

For further details see British Patent No. 229,725.

Function of Water in Wet Spinning of Flax

Flax which has been retted in dew behaves in a different manner from that which has been retted in water. If as low temperatures as possible are employed in the process, in order to bring into contact with flax fibers as little as possible soluble pectin in the form of a viscous solution, then the fibers slip very readily. Flax which has been retted with the aid of water contains considerably less soluble pectin, and hence the spinning is carried out at a higher temperature, and a corresponding absorption of water must be effected by the insoluble pectin. In both kinds of flax, that is, flax retted with water, efforts should be made to obtain as high as possible absorption of water by the pectin compounds, and at the same time as little as possible loss of pectin compounds by solution in water. (Farben Zeitung, volume 20, pages 314-7.)

Cleansing Vegetable Fibers

Various vegetable fibers, such as flax, hemp, ramie and the like are treated for a period of 2 hours in a bath which consists of water, glycerine and chloride of lime. The chloride of lime is conveniently suspended in a basket or container of some sort in the bath. After the fibers have been washed with water, they are heated to a temperature of 120 deg. Cent. in a closed apparatus with an aqueous solution of sodium hydroxide, chloride of lime and hydrochloric acid. After the fibers have been allowed to cool, they are washed with an aqueous solution of an oxidizing agent, such as hydrogen peroxide, hypochlorite. Only a small amount of the oxidizing agent is used for this purpose. The fibers are then washed once again and dried and finally passed between heated rolls. The fibers thus obtained are then combed and are ready for spinning. (French Patent No. 573,684.)

Dyeing Wool

Wool is dyed by immersion in aqueous suspensions of mono-, di- or tri-amino anthraquinones, alkylaminoanthraquinones, or substituted derivatives of these substances, but excluding hydroxyl derivatives or related acid dye-

stuffs, dispersing agents such as alcohol and cresol being added.

For example, 100 pounds of woolen yarn are thoroughly wetted down, then dyed at a temperature of 90 deg. Cent. for a period of 30 to 45 minutes in a volume of 200 to 400 gallons of water which contain from 2 to 4 pounds of alcohol, and 1 pounds of 1-amino-2-methylanthraquinone. The wool is afterward rinsed for the removal of undissolved dyestuff, and then dried. Suitable dyestuffs are 1-methylaminoanthraquinone, 2-aminoanthraquinone, 1-chloro-2-aminoanthraquinone, 1:4-diaminoanthraquinone, etc.

DYESTUFF TABLES

(*Continued from page 439*)

DISCHARGING: Zinc discharges the color white.

COMPETING PRODUCTS: Made in the United States by Albert David Chemical Company, under name of Spirit Blue.

ACID MAGENTA

(Schultz No. 524)

COMPOSITION: Triphenylmethane.

SPECIALLY SUITABLE FOR: Wool.

USUAL METHOD OF DYEING: Glauber salt.

SHADE: A very brilliant full and bloomy shade.

SHADE BY GASLIGHT: Unchanged.

LEVEL: Dyes level with care.

FASTNESS TO:

Acid: Fast.
Alkali: Slight.
Carbonizing: Fast.
Light: Very poor.
Perspiration: Very poor.
Rubbing: Fast.
Steaming: Does not stand steaming.
Street Dirt: Very poor.
Sulphur: Goes yellower.
Washing: Slight.
Water: Slight.

DYED BY OTHER METHODS: Stands chroming.

ON UNIONS: Dyed acid, cotton-effect threads are only slightly tinted. On gloria: Dyed in strongly acid bath and given a hard boil, the silk is hardly tinted.

ON OTHER MATERIALS: On silk: Dyed in bath of boiled-off liquor and sulphuric acid. Suitable for straw and feathers. One of the few colors that will dye living flowers.

DISCHARGING: Suitable for color discharges with tin. On silk: Hydrosulphite gives a white discharge, but the color returns on storing.

COMPETING PRODUCTS: Made in the United States by Cincinnati Chemical Works, under name of Acid Magenta; Newport Chemical Works, under name of Acid Magenta M.

ACID VIOLET 4BN

(Schultz No. 527)

COMPOSITION: Most acid of the acid violets.

SPECIALLY SUITABLE FOR: Wool.

USUAL METHOD OF DYEING: With Glauber salt and sulphuric acid. (If added to an acid bath the bath must be boiling.)

SHADE: Reddish violet; much redder than S-530.

SOLUBILITY: Poor; add a little soda.

LEVEL: Dyes level.

FASTNESS TO:

Acid: Fast.
Alkali: Moderate.
Carbonizing: Stands carbonizing.
Fulling: Not suitable; shade goes lighter. Whites are stained.
Ironing: Fast.
Light: Moderate; inferior to S-530.
Rubbing: Crocks off.
Steaming: Fast.
Street Dirt: Fast.
Sulphur: No change in shade; bleeds slightly.
Washing: Scours off.
Water: Bleeds off in hot water.

DYED BY OTHER METHODS: Can be dyed chromate or chrome mordant. On a chrome mordant the shade is bluer.

ON UNIONS: Wool-silk unions: With sulphuric acid, both fibers dyed equal. Cotton-wool unions: Dyed acid, the cotton is slightly bluish. Wool is dyed a full shade in a neutral bath.

ON OTHER MATERIALS: Silk: Dyed in bath of boiled-off liquor and sulphuric acid. Suitable for felt hats on account of good penetration.

DISCHARGING: Zinc discharges the color white. Tin gives a colored discharge with this color.

COMPETING PRODUCTS: Made in the United States by Chemical Company of America, under name of Acid Violet 4BN; National Aniline & Chemical Company, under name of Erie Violet 3R.

The Supreme Court of the United States has granted the motion of the Solicitor-General advancing the date for hearing the appeal from the decision of the United States Circuit Court of Appeals in the Chemical Foundation case. The case has been set for Monday, November 2nd, and argument will be held after the cases heretofore assigned for that date.

At Cleveland, Ohio, the Industrial Fiber Company, manufacturers of rayon, will begin at once the erection of three additions to their plant, at a cost of $300,000. There will be a finishing building and a textile building, each of saw-tooth construction, and an acid building of three stories, fireproof. A number of alterations will also be made in the present plant.

OXYCELLULOSE AND ITS TESTS

(*Continued from page* 419)

be obtained, of course, if the material has been alkali boiled in process during or after oxidation, so it is most useful as a confirmatory test.

High ash alkalinity, remaining after a standard washing of the cotton, is characteristic of those oxycelluloses that have a high Methylene Blue absorption, and is apparently unrelated to the type that results in high copper number.

The Older Qualitative Tests

The large number of oxycelluloses prepared in the above work has also been used to investigate some of the older qualitative tests, and since several of these methods have been found to be characteristic of one or the other of the two types of oxycellulose they are found not to be individually sufficient as criteria of oxidation.

Three tests that depend on the reducing properties are: Staining test with silver solutions; staining test with Nessler solution; dyeing test with Indanthrene Yellow. Of these, the first two are somewhat related to the reduction of Fehling's solution, and they readily detect the oxycel'uloses with the high copper numbers, while failing with the Methylene Blue type.

The fixation of Indanthrene Yellow from a hot alkaline suspension, on cotton containing oxycellulose, is an interesting test in which the reducing properties of the latter are made to serve the purpose of hydrosulphite. "It illustrates one of the practical disadvantages of the presence of oxycellulose in cotton fabric; namely, the bleeding or running onto a white ground which may take place in an alkaline treatment from colored stripes containing vat dyes." The test is sensitive for oxycellulose of copper number down to 0.5.

Two other tests that respond to the copper oxycellulose but not to the Methylene Blue type are: Color test with phenylhydrazine; color test with Schiff's reagent. Neither of them is sensitive.

The tendency of cotton goods containing oxycellulose to go yellow on heating or on treatment with alkali is intimately connected with the reducing properties, or high copper number. Oxycelluloses were found which had suffered severe tendering, but did not respond to either yellowing test. They were of the Methylene Blue type.

Behavior with direct dyes is not a very sharp test, but is more suitable for the type of oxycellulose with high Methylene Blue absorption.

Practical Interpretation

The yellowing of oxidized cotton in storing bleached goods is associated with an enhanced copper number, and the latter may be considered a test for permanence of white, it being understood, however, that other causes may contribute to yellowing.

In various technical bleaches, chemicking was accompanied in every case by a significant rise in copper number, but bleaches have been obtained by a very thorough boil and light chemic, where a good white was unaccompanied by any significant increase. Full bleaches usually give copper numbers ranging from 0.01 to 0.3. A tentative maximum for good bleaching is suggested as 0.25 to 0.3.

From a practical point of view, the Methylene Blue may be considered as a test for level dyeing properties. If this quantitative test gives results uniformly high at different places in the lot, even dyeing may be expected, but the higher it is the more chance there is of unevenness.

The loss of viscosity is not directly due to the presence of cellulose oxidation products, but presumably to an ultramicroscopic change in the structure of the residual cellulose, which produces a decrease in the strength of the individual cotton hairs. Although there is as yet no known quantitative relation, the test should be interpreted as indicating tendering of the cotton.

The values of viscosity in "2 per cent cuprammonium solution" in absolute units generally lie between 2 and 3 for "scoured" cotton and between 1 and 2 for fully bleached. The value 1 may be tentatively fixed as the minimum permissible in a good bleaching process, and it is improbable that significant tendering has resulted down to this point.

"The test of successful bleaching is the attainment of good white *without sacrificing other textile qualities*, and in judging the success of a bleaching process the whiteness of the material should be the last though not necessarily the least important consideration. If a bleached material has proved satisfactory with respect to certain chemical standards such as those suggested above, then the efficiency of the process may be judged by the purity of white."

CAMPBELL ISSUES ARTIFICIAL SILK CARD

A new card, handsomely bound, showing 86 standard colors for dyeing Viscose silk, has been issued by John Campbell & Co. The colors, displayed neatly in four panels, comprise shades from this manufacturer's Amidine, Sol-Amidine, Amalthion and Basic series of colors and range from brilliant hues of yellow and orange to lustrous blues and blacks. A folder attached to the shade card gives detailed dyeing directions for each series. Dyers of Viscose silk will find this new card a timely addition to their color card file.

The Summit Bleaching & Dyeing Company has been chartered with capital of $100,000, to take over and operate the plant of the Summit Dyeing Company at North Bergen, N. J. Arthur Weise, Adolph Scherer and William B. Rocchi head the new company.

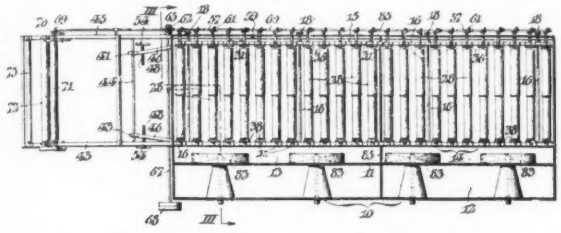
RECENT PATENTS

Drying and Tentering Machine

(1,531,826; March 31, 1925)

Harry H. Wigglesworth, Philadelphia, Pa., assignor to the Philadelphia Drying Machinery Company, Philadelphia, Pa.

A machine of the character described, comprising an enclosure subdivided by longitudinal partitions into



drying and heating compartments with an intermediate thoroughfare, means to progress material of different widths through the drying compartment in a horizontal direction along a path composed of vertical undulations, and means for tentering the material on the progressing means while drying.

Brown Sulphur Dyestuffs

(1,531,925; March 31, 1925)

Louis Hass, Paris, France, assignor to the Societe Chimique de la Grande Paroisse, Paris, France.

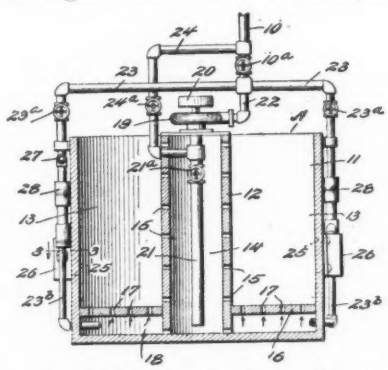
A process for the manufacture of brown coloring products having a red tinge consisting in treating crude trinitronaphthol with a polysulphide of an alkali metal.

Dyeing Apparatus

(1,531,997; March 31, 1925)

Francis C. W. Stelter, Long Island City, N. Y.

A dyeing apparatus comprising a tub having tangentially arranged inlets in its side walls, a centrally



arranged well within the tub, the well being in communication with the surrounding space within the tub, and a pump and suitable piping arranged to withdraw dye liquid from the well and deliver it into the tub through said inlets whereby the liquid may be continuously circulated and the goods under treatment agitated, for the purpose set forth.

Waterproofing of Fabric Articles

(1,535,999; April 28, 1925)

Arthur George Harrington, London, England, assignor of one-half to Harold Lionel Brandon, London, England.

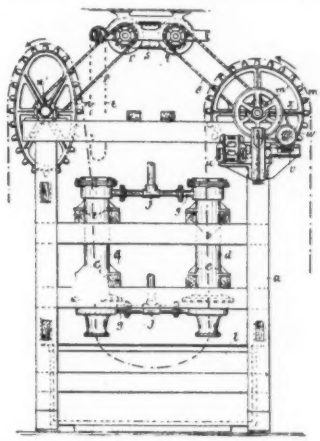
A composition for waterproofing fabrics including hard paraffin wax dissolved in petrol to which a small proportion of trichlorethylene has been added.

Apparatus for Treating Fabrics with Liquids

(1,535,251; April 28, 1925)

Clifford H. Ramsey, Glen Rock, and Arthur C. Maskery, Paterson, N. J., assignors to Morrison Machine Company, Paterson, N. J.

In combination, a pair of passaged conductors having their passage axes upright and being adapted to receive the upstanding portions of the depending bight



of the flexible attenuated material to be advanced longitudinally and each having means to admit to its passage liquid to treat the material while being advanced, and separate means to advance said material arranged over said conductors and from which said bight hangs, one of said means arranged to deliver the material down to one conductor and the other to draw it up from the other conductor.

Art of Reducing Indigo Dyes and Other Solutions

(1,535,151; April 28, 1925)

Frederic A. Eustis, Milton, Mass.)

The art of reducing an indigo dye solution which comprises mixing a part of the dye solution to be reduced with zinc dust, adding sulphur dioxide to the mixture to form zinc hydrosulphite, adding an alkaline agent to the solution to convert the zinc hydrosulphite and other zinc salts into sodium salts, and returning said sodium salts in solution to the rest of the dye solution.

FALL SHOE AND HOSIERY COLORS

In a supplement to the 1925 Fall Color Card just released for general distribution by the Textile Color Card Association there is contained the six shoe and leather colors selected by the joint Color Committee of the Allied Shoe and Leather Association.

These colors will be found to harmonize with many of the new silk and wool shades, as well as the hosiery colors recently issued. This is a result of the close co-operation of the various committees in their selection of colors for fall and winter.

These colors include Autumn Blond, India Tan, Rugby Tan, Woodland Brown, Rosewood and National Gray.

The first seasonal Color Card for Hosiery has also been issued by the Textile Color Card Association in co-operation with the National Association of Hosiery and Underwear Manufacturers. Supplementary to the Standadr Hosiery Color Card, the 125 Fall Season Hosiery Color Card contains twelve colors selected by the official committee appointed by the joint associations.

Many of the colors recommended for fall were chosen from the Standard Hosiery Card. The range is characterized by pale neutral shades which portend the continued popularity of light hose.

The twelve colors shown in silk are, Almond, Atmosphere, Aztec, Bran, Champagne, French Nude, Gunmetal, Mauve Taupe, Moonlight, Piping Rock, Rose Gray and Rosewood.

The Hosiery Committee in the selection of the fall colors worked in conjunction with the Shoe and Leather Committee and the allied Shoe and Leather Associations. They also tempered their selection by the colors featured for wool and silk.

The co-operation of the various committees representing all branches of textile industries and related trades is a step of far-reaching significance and in response to the present trend of fashion and increasing importance of color.

PONTAMINE SCARLET B

The production of a direct dyestuff known as Pontamine Scarlet B is announced by the Dyestuffs Department of E. I. du Pont de Nemours & Co. It is stated to yield brilliant scarlets of a somewhat bluish shade and to be suitable for dyeing all classes of cotton goods, such as raw stock, yarns and pieces, and due to its very good solubility may be employed in the padding machine.

It is described as particularly adapted for use on pure silk on which bright shades may be obtained from a soap bath broken either with acetic acid or the mixed acid that is in general use in the silk mills, or from a neutral or acid bath. It may also be used to good advantage in the garment dyeing trade on fabrics of any description as a self-color as well as a base for such shades as bright reds, Bordeaux and wine shades.

At Providence, R. I., the U. S. Finishing Company has taken out a building permit and will start work immediately on a one-story dyehouse addition.

GRASSELLI SALESMAN HAS ANNIVERSARY

Charles A. McNeil, salesman in charge of the Leather Department of the Grasselli Dyestuff Corporation, 117 Hudson Street, New York City, was tendered an impromptu reception by his associates on the occasion of his 25th anniversary with the company on June 15, 1925.

As an indication of the friendly spirit prevailing between the Grasselli Dyestuff Corporation and its employees, Mr. McNeil was presented with a handsome set of silverware on the part of the company, as well as a leather traveling bag and basket of beautiful cut flowers by his associates and friends.

In reply to the demand for a speech, Mr. McNeil gave a very interesting discourse on the many changes which have taken place in the American leather industry during his many years of eventful service.

WILLIAM ARMSTRONG

William Armstrong, a director and for many years superintendent of the Hamilton Cotton Company, Ltd., Hamilton, Ontario, died on May 25. Mr. Armstrong was with this company from the time of its inception in 1880.

The Weidmann Silk Dyeing Company, Lodi, N. J., has awarded a general contract for a one-story and basement dyehouse on Fifth Avenue, Paterson, to be 61 by 200 feet, and to cost about $75,000 with equipment.

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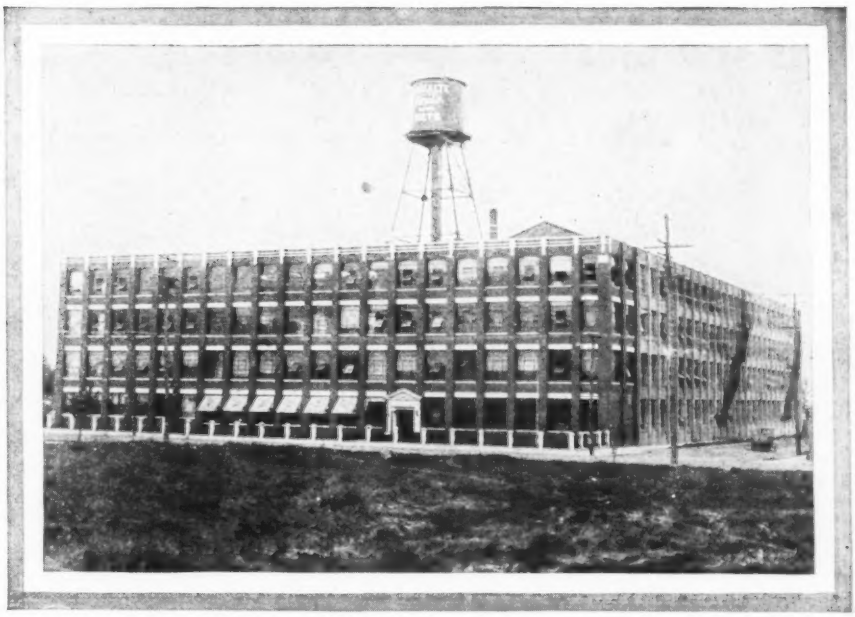
WANTED—College graduate with experience to teach Textile Chemistry and Dyeing. Address Box 307, American Dyestuff Reporter.



Plant of E. & Z. Van Raalte, Paterson, N. J., that has softened its water supply with a Permutit Water Softener since August, 1914. Permutit Softeners are also installed in the Van Raalte plants at Saratoga Springs and North Tonawanda, N. Y.

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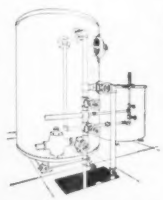

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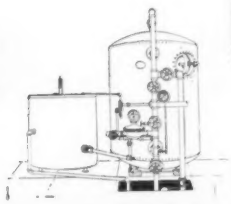

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in the English Language

Edited by

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assisted by
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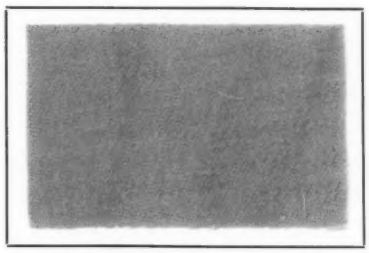

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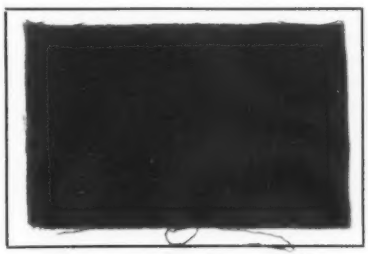

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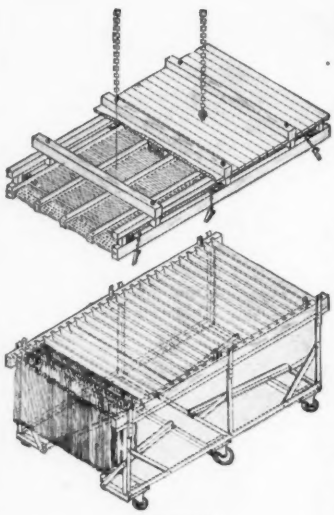

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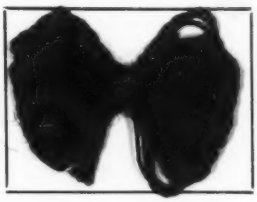

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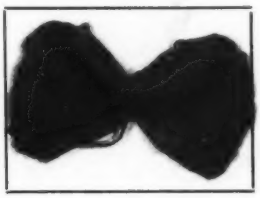

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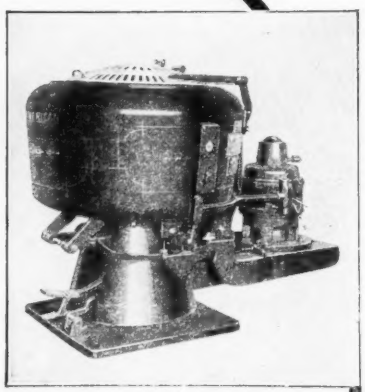

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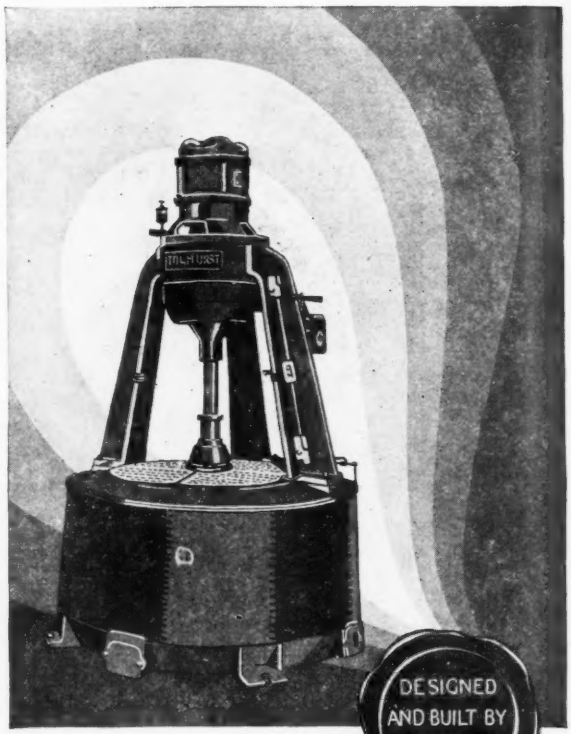

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Devoted to the practical application of dyestuffs in all industries; especially to textile chemistry—the science and practice of scouring, bleaching, dyeing and finishing.

NEW YORK, JULY 13, 1925

Wool-Scouring Waste Liquors—Composition and Disposal

Treatment of Waste Scouring Liquors—Experimental Work by the Bureau of Chemistry—Wool Grease—Laboratory Experiments to Obtain a Concentrated Waste Liquor—Fertilizer Materials from Waste Liquors—Value of Recoverable By-Products

By F. P. VEITCH and LEON C. BENEDICT

THE disposal of wool-scouring waste liquors is one of the most important and difficult problems with which industry and chemical engineers are confronted. Wool-scouring waste liquors are exceptionally complex. They contain natural wool fats and waxes, animal exudations and excrement, and large quantities of dirt, loaded with bacteria, to which are added soaps and alkalies, the whole making an emulsion that is exceedingly difficult to break in any practical way and that soon clogs and puts out of commission any known purifying filtering system. On the basis of the figures obtained in the work outlined in this paper, it is estimated that the wool-scouring waste liquors of this country contain from sixty to seventy million pounds of grease, sixty to ninety million pounds of dirt, forty to forty-eight million pounds of potash salts, expressed as potassium sulphate, and fifteen million pounds of nitrogenous matter, expressed as dried albumen. In marketable form these would be worth approximately $5,000,000.

Without question, the object of any wool-scouring plant is to produce a thoroughly cleansed and high-quality product at the lowest cost. During the early years of wool scouring the waste liquors received but little attention, if any, and were emptied directly into the streams, regardless of their polluting effect on the water supply. As it comes from the scouring machines the waste liquor is very dirty, greasy and foul smelling, and in the course of a few hours is rapidly decomposed by fermentation, thus liberating the foul, slimy matter contained in it. This in turn is deposited on the bottom and along the banks of the streams. As these streams, either directly or indirectly, supply the drinking water for many of our villages and big cities, it can be readily seen that sooner or later legislation must be passed to prohibit the introduction of such obnoxious matter. In fact, such legislation exists at the present time in a number of the Eastern States. The many wool-scouring plants which are not yet subject to these laws, however, turn their waste liquors directly back into streams without giving them any purification whatsoever. From the sanitary point of view, these liquors are a most objectionable source of pollution to any water supply. From the industrial point of view, such polluted waters are a serious source of annoyance and expense to all plants which have been built farther down the rivers. In fact, many desirable factory sites have been abandoned because of the pollution of the water supply by other industries.

It is generally recognized that streams into which all the waste liquors, either unpurified or partially freed from grease, are allowed to run are so highly polluted that they are not suitable for household use or for many industrial purposes. We shall, therefore, discuss the pollution itself no further, but consider briefly the methods of purification of the waste liquors and the utilization of the wastes. How may such pollution of our water supplies, with the attendant danger to public health and damage to industry, be avoided, and how may these wastes be put to some economic use? We can not, of course, offer a new solution of this problem, which has been studied by many investigators from practically all angles. Our aim is rather to emphasize the significance of what has been done, in the hope that it may promote a more complete and practical study than has yet been made and a more satisfactory consideration of the numerous processes and types of installation which have been developed to meet this need.

Treatment of Waste-Scouring Liquors

Since only a small part of the six to seven hundred million pounds of wool scoured each year in this country is scoured by the solvent process (to be exact, only two

mills are using this process), the waste liquors from the soap-water scouring methods present the most serious problem. For many years it has been known that spent scouring liquors contain by-products which, if they could be recovered economically, would be commercially valuable; in fact, a large number of wool-scouring mills have installed plants to recover one or more of these constituents.

The more important of the methods which have been tried with waste-scouring liquors are: (1) The "acid-cracking" process, one of the first to be employed and still widely used both here and in Europe; (2) the "battage" process (French patent No. 413,442); (3) the treatment of waste liquors with gases, such as sulphurous-acid gas; (4) the steeping process for the concentration of potash, followed by the acid treatment for grease recovery; (5) the Cardem process, in which, after the grease is removed, the waste liquor is reduced to a dense liquid or to a powder by spraying it into a heated chamber; (6) solvent extraction of grease, followed by potash recovery; (7) the evaporation of the liquors to a small volume and subsequent centrifuging to recover the grease; (8) the clarification of the liquors by centrifuging and the re-use of the centrifuge liquor for scouring more raw wool, thus increasing its potash concentration; (9) centrifuging the settled, heated waste liquors for the recovery of the greater part of the grease, which is of a much higher grade than "degras," and running to waste the partly degreased waste liquor. Of these, Nos. 1, 4, 5, 7, and 9 seem most useful or have been widely used. None of the others will be considered here.

The acid-cracking process has been most generally used, owing no doubt to the fact that its installation does not require a large outlay of money and it can be operated fairly successfully with unskilled labor. The process consists chiefly in treating the waste liquors with sulphuric acid until they are slightly acid, then agitating the mixture by means of air blasts, allowing the sludge to settle, removing and running to waste the intermediate acid-water layer, collecting the agglomerated grease and dirt, together with the fatty acids from the soap, and subsequently recovering the fats and grease by treating the sludge in hot filter presses. The recovery of the fats and grease is fairly complete (usually between 95 and 98 per cent). The recovered grease is of a very low grade, however, and is usually marketed under the trade name of "degras." In the acid-cracking process the potash salts and nitrogenous materials are lost, and the resulting acid liquor containing these constituents is difficult to purify further and cannot be discharged into streams without seriously polluting them.

The steeping process, followed by the acid treatment for the recovery of the grease, has been used in England, France and Belgium, as well as in the United States. The object of this method is to obtain a *concentrated* potash liquor, from which potash salts can be economically recovered. In view of the fact that the potash salts on the raw wool are largely soluble in cold water, at which temperature the wool grease is but slightly affected, the usual method is to steep the raw wool in cold water, the tanks (commonly two or three) being arranged according to the counter-current system, so that the liquor moves in one direction and the wool in the opposite direction. When the concentration becomes high enough, the liquor is condensed to approximately one-tenth its original volume in triple effect evaporators, after which it is incinerated in a reverberatory furnace, producing a red-brown coke containing approximately 40 per cent of potassium oxide. This material is used in making fertilizers, especially for tobacco, where potash in the form of the carbonate is often preferred. The partially washed wool is then scoured in the usual manner to remove the residual grease, and the waste liquor is treated for the recovery of the grease by the acid-cracking process.

The steeping process has met with disfavor because it requires expensive special apparatus and, unless the process is continuous, the liquors become foul and practically unworkable long before the desired potash concentration is reached. Furthermore, the nitrogenous material contained in the liquors is destroyed. Before the World War, the largest recovery and purification plant of this type was in Bradford, England. After the United States entered the war a large plant was erected at Lawrence, Mass., for the recovery of potash salts from waste-scouring liquors. In this plant the ordinary waste-scouring liquors from the first and second bowls were heated and passed through high-speed centrifuges. It was stated that this treatment removed approximately 70 per cent of the grease. The partially degreased waste liquor was evaporated, the solids were charred in rotating kilns, leached, and the potash salts recovered. When the price of potash was high, this plant was able to operate at a profit, but directly after the signing of the armistice it ceased operations, indicating that this method of treatment cannot be considered a commercial success when potash sells at its normal price of approximately $40 to $50 per ton for muriate or sulphate.

The solvent-scouring process, known also as the solvent process, consists of first extracting the grease with a volatile solvent (such as commercial naphtha) and then washing the degreased wool with warm water to remove the suint or potash salts. Only two mills in the United States employ this process to-day. Of the several objections which have been advanced to this process probably the most serious is that the scoured wool looks dead. Among its advantages are lower cost of scouring, easier recovery of potash salts, wool grease of a good grade, and material decrease, if not complete elimination, of stream pollution. Several patents have been granted on this process, but the methods and apparatus employed differ only slightly. Briefly it is as follows: The kier or extraction tank is filled with wool (approximately 1,000 pounds to a charge) while the movable false bottom, which is attached to the hydraulic ram, is at the bottom of the kier. The cover is then securely fastened on the kier, and the solvent is forced through the wool and allowed to remain in contact with it for from 15 to 30 minutes, after which the false bottom is raised and the

solvent pressed out and withdrawn through specially arranged pipes. A second and third treatment with solvent now follow, after which the degreased wool is washed once or twice with warm water, removed from the kiers, and transferred to the ordinary soap and water scouring machine, where it is given a final *brightening* scour with a very dilute solution of soap, usually less than 0.1 per cent. This treatment removes the suint and dirt and is said to impart a more "lofty" and brighter appearance to the finished product.

The composition of the material at various stages of the commercial solvent scouring process is shown in Table 1.

TABLE 1

Composition of Sludge and Wash Water from Solvent Probess

Determination	Sludge Which Settles to Bottom of Tark Containing Gasoline Extract from Raw Wool Before Renewal of Gasoline	Sludge Which Settles to Bottom of Tark Containing Gasoline Extract from Raw Wool After Renewal of Gasoline	Wash Water Obtained by Washing Wool After Gasoline Extraction
Total solids	34.3%	21.0%	2.1%
Ash	19.2	10.6	1.1
K_2O	4.2	2.5	0.6
Total nitrogen	0.7	0.4	0.1
Calculated to Dry Basis			
Ash	56.0	50.5	50.7
K_2O	12.1	11.8	28.8
Total nitrogen	2.0	1.8	3.2

The raw wool scoured by this process usually contains a small percentage of water, as does also the solvent used. This accounts for the presence of the potash in the gasoline extract. Ordinarily potash salts are insoluble in gasoline, but, owing to their solubility in water, they are partially removed with the solvent-grease-water solution.

Smith-Leach Process.—The scouring liquor is evaporated to a small volume, centrifuged to remove grease, and subsequently evaporated and incinerated as described in the "steeping" process. Wilson ("Trade Waste Waters") states that from 55,000 gallons of raw scouring liquor two tons of wool grease and one ton of potassium carbonate can be recovered.

Centrifuging the settled, heated waste liquors for the recovery of a high-grade wool grease is a process that was developed rapidly in this country during the World War and, with the acid-cracking process, is the one most generally used here. The spent scouring liquors are first run into settling tanks for the removal of heavy materials, such as sand and dirt, and then through heating coils into high-speed centrifuges, where a large portion of the wool grease is removed. The resulting liquor, although containing much grease and other impurities (largely nitrogenous) derived from the raw wool, is emptied directly into the streams. The recovered grease is of fair quality and finds extensive uses in the manufacture of cosmetics and many other articles. However, this process falls far short of needed effectiveness both in purification of the liquors and in the recovery of their components.

Experimental Work by the Bureau of Chemistry

Search of the literature showed that the information on the composition of the impurities present on raw wool of various types and grades and on the quantities of such impurities and also on the composition of the waste liquors in which known wools had been scoured was fragmentary and unsatisfactory. For this reason we undertook the analysis of a large number of wools, endeavoring to have a sufficient number of samples of each type to establish with reasonable certainty the maximum, minimum and average percentages of the more important constituents present in each type. On some types we fell somewhat short of our aim.

Whenever possible, a member of the Bureau selected the samples, thus making sure that they were reasonably representative of the raw wool or the waste scouring liquor, as the case might be. These samples were shipped immediately to Washington, where they were carefully subsampled before analysis. The plan of procedure for raw wool was as follows: Moisture was determined at 100 to 105 deg. Cent. The sample was then completely

TABLE 2

Impurities in Wool of Various Kinds

(*Based on Wool as Analyzed*)

Grade	No. of Samples	Moisture	Pet. Spirit Extract Max.	Pet. Spirit Extract Ave.	Pet. Spirit Extract Min.	Total Ash Max.	Total Ash Ave.	Total Ash Min.	Water Extract Max.	Water Extract Ave.	Water Extract Min.	K_2O in Water Extract Max.	K_2O in Water Extract Ave.	K_2O in Water Extract Min.	Nitrogen in Water Extract
Fines	93	5.6%	41.9%	17.4%	6.1%	47.6%	23.8%	8.7%	22.9%	14.0%	6.3%	6.5%	3.6%	1.1%	0.51%
½ Blood	48	5.5	27.7	12.7	5.9	36.8	23.0	10.0	22.6	15.2	10.6	5.7	3.9	2.0	0.57
Medium	29	5.0	16.6	10.5	5.5	46.8	19.0	8.9	27.4	14.7	8.8	7.0	3.6	1.3	0.48
⅜ Blood	44	6.3	16.3	9.8	6.7	38.0	19.4	9.7	33.0	16.8	8.1	5.9	4.5	1.9	0.60
¼ Blood	38	7.0	15.0	8.6	6.4	34.5	18.3	9.7	25.7	15.4	6.1	7.3	3.9	1.4	0.52
South American miscellaneous.	14	6.2	15.1	11.3	7.6	42.1	23.1	8.9	22.3	13.8	7.2	5.9	3.5	1.3	0.27
Miscellaneous	29	6.4	23.9	12.8	5.7	35.1	22.8	5.9	19.8	14.8	3.6	5.9	3.8	0.2	0.54
Pulled	10	3.5	19.4	10.8	5.1	19.8	8.9	3.7	5.1	3.5	1.7	0.7	0.2	0.1	0.31
Total	305	5.87	—	12.87	—	—	21.22	—	—	14.55	—	—	3.77	—	0.52

degreased in a Soxhlet extractor, using petroleum spirit as the solvent, the resulting solvent-grease solution was evaporated, and the residual grease was determined. The degreased sample was extracted in the same apparatus with boiling distilled water and the water extract was taken to dryness, thus giving the total water-soluble extract. This residue from the water extract was again dissolved in distilled water and made to known volume. An aliquot was evaporated to dryness and finally ashed to constant weight, thereby affording a means of ascertaining the relative percentages of organic and inorganic matter which had been extracted by the distilled water treatment. The potash (K_2O) content of the water extract was then estimated on another aliquot, according to the methods of the Association of Official Agricultural Chemists for fertilizers. A third aliquot portion of the water extract was used for the determination of the nitrogen content by the regular Kjeldahl method. Finally the total crude ash content of the raw wool was estimated on a thoroughly dried sample of the original raw wool by burning to constant weight in an electric muffle.

TABLE 3
MEAN GREASE AND POTASH CONTENT OF RAW WOOLS BY LOCALITY

Locality	Number of Samples	Pet. Spirit Extract	K_2O in Water Extract
United States	213	12.74%	3.98%
South America	33	10.36	3.42
Cape	20	16.14	3.90
United States (pulled wool)	10	10.83	0.24
Australia and New Zealand	22	15.50	3.19
Miscellaneous	7	13.15	4.20
	305	12.85	3.80

Tables 2 and 3 show the maximum, minimum and average results of analysis of some similar 305 samples of various wools in the grease. A careful examination of these figures should afford information directly applicable to the problem of treating waste-scouring liquors. Speaking broadly, the average wools being scoured in this country will probably yield 13 per cent of grease, 4 per cent of potash (K_2O), ½ per cent of nitrogen, 21 per cent of ash, and 14½ per cent of water-soluble matter of which about 3 per cent is nitrogenous matter. These averages, however, can be used only in a general way. Should data on the individual wools of any one type or grade be desired, reference should be made to the results published elsewhere. It is important to note that the average figures for the water-soluble potash content of wool of different kinds or of wools from different countries do not differ widely, except, of course, in the case of pulled wools, although the grease content may differ materially with the kind of wool being scoured.

No attempt has been made in the tables to effect more than a general classification of wools. The data on individual samples will be published later. The wide variation in grease and water-soluble potash content of different samples of the same kinds of wool, however, is interesting, especially as both are to a large extent governed by genetic and geographical conditions. For example, the sample of fine wool, which contained approximately 42 per cent of grease, was grown in Ohio under ideal genetic and grazing conditions and was practically free from dirt and sand, whereas the sample containing only 6 per cent of grease was grown in Texas, where the sod was so scanty that the fleeces were heavily contaminated with fine red sand and vegetable matter. Both of these wools would shrink about to the same extent but obviously they would require different handling in scouring and their waste-scouring liquors would differ materially in composition. This is mentioned merely to bring out the exceedingly wide variation which may be found in wools of the same grade

WOOL GREASE

As is well known, the most objectionable, and from an industrial point of view the most detrimental, constituents of waste scouring liquors are the wool grease and decomposed soaps. Wool grease is an exudation from the body of the sheep, which serves the purpose of a protective coating from the wool fiber during its growth. It consists for the most part of higher alcohols and therefore has the properties of a wax rather than those of a true grease. As pointed out by Andes ("Animal Fats and Oils"), crude wool grease is a greasy yellow or brown mass, with an unpleasant smell, containing a large quantity of cholesterin, isocholesterin, stearic and palmitic-cholesteryl-esters, which make it imperfectly saponifiable by alkalies. For this reason and also because of its insolubility in water, the method employed in removing it from raw wool is one of emulsification. The waste liquors, therefore, contain the natural wool grease very closely emulsified with the scouring agents used, as well as with the potash salts present on the raw wool.

Of the numerous methods proposed for the recovery of wool grease, only the following three are used today in this country—the acid-cracking process, the centrifuge process, and the naphtha solvent extraction process. The selection depends to a large extent upon State laws relating to the pollution of streams, the type of wool scoured, and whether "degras" or "neutral wool" grease is to be recovered.

Scouring mills in States which have rather rigid laws governing stream pollution usually employ the acid-cracking method. As recovered by this treatment the grease is a brown-black, greasy mass, with an unpleasant smell, containing true wool grease, fatty acids from decomposed soaps, fatty acids from the original wool grease, inorganic salts, which were either present on the raw wool or were added during the scouring, decomposed albuminous and glutinous matter, and a fraction of one per cent of sulphuric acid.

Some investigators believe that this material can be purified and converted into "lanolin." The Bu-

reau's attempts to do this have been unsuccessful. It appears almost impossible and clearly impracticable to accomplish this purification on a commercial scale. The relatively high temperature employed in the process of pressing out the grease decomposes the albuminous matter, forming exceedingly stable dark compounds, which in turn are so closely mingled, if not actually combined with the true wool grease, that the strongest bleaching agents have but a very slight effect upon the grease. The conclusions, therefore, are that more than a partial purification of recovered grease of this type is very costly and commercially impracticable.

The centrifuge process gives much better and more uniform grease, which with proper treatment can be converted into either "neutral wool grease" or "lanolin." The industry finds that best results are obtained when the waste-scouring liquors, after thorough settling, are heated to approximately 190 deg. Fahr. and conducted directly into high-speed centrifuges, operated under 35 to 40 pounds of steam pressure and at a speed of from 15,000 to 17,000 r.p.m. As it is delivered from these machines the grease is of a medium brown color, has a slight odor of sheep and soap, contains between 4 and 5 per cent of water, and possesses an acid value of from 3.5 to 4. It is the custom in some mills, which scour only high-grade wools, to recentrifuge this grease, which gives a product containing less than 1 per cent of water and having an acid value of from 0.5 to 1. This product finds a ready market with any lanolin manufacturing establishment and is quoted at from $0.04 to $0.07 per pound.

Naturally the composition of the degreased waste liquors coming from the centrifuge varies considerably. Such a liquor from scouring wool showed: Ash, 1.03 per cent; potash (K_2O), 0.81 per cent; grease (as wool grease), 0.08 per cent; fatty acids, 0.82 per cent; nitrogen, 0.08 per cent, and total solids, 2.93 per cent.

Because of its low grease content, such waste liquors can be treated for the recovery of the fatty acids and potash at a decidedly lower cost than can the original liquor carrying the grease. It was observed that the greases recovered from the waste liquors from the better-grade wools were much lighter in color and contained a smaller percentage of free fatty acids and nitrogenous matter, indicating that different recovery methods may be advisable for scouring liquors from wools of different types and grades.

The results of our experiments on greases recovered from commercial waste-scouring liquors indicate that the main constituents of such greases are free fatty acids, from the wool fat proper, neutral saponifiable matter, consisting of free alcohols, such as cholesterol and isocholesterol. Purified wool fat may be said to consist mainly of two portions, one, melting at approximately 60 deg. Cent., which is insoluble in alcohol and also unsaponifiable, and another, melting at approximately 15 deg. Cent., which is soluble in alcohol and also saponifiable.

Wool grease is used, either in its crude or refined state, in soap manufacture, rubber manufacture, leather manufacture, hoof salves, rust preventives, improving oils poor in fats, as a substitute for tallow, palm oil and castor oil, mixed with tallow, graphite, etc., for locomotives, in wire drawing oils, wire cutting oils, rope and cordage manufacture, in lanolins, ointments, cosmetics and shaving creams, and in sticky mixtures for catching insects, lithographic and multographic inks, calico printing inks, shoe blackings, wool oils for carding and combing wools, special varnishes, and quenching oils, for waterproofing and coloring electric bulbs, special cylinder oils, stop-cock greases, pyroxylin coated fabrics, foam breakers, waterproofing fabrics, etc., and as a lubricant in steel rolling.

The annual production of wool grease in this country has been estimated to be between 40,000 and 50,000 barrels; the consumption averages approximately 70,000 barrels; thus nearly half of the wool grease used here must be imported. The imports for January, February and March of this year through the ports of Boston, New York and Baltimore alone amounted to over 3,100 barrels per month. These figures show an urgent need for the development of some economical and adequate method for treating waste-scouring liquors for the twofold purpose of recovering the grease and eliminating one of the most serious sources of stream pollution.

Although waste-scouring liquors contain only about half of one per cent of nitrogen, the nitrogenous matter (not including that which settles out with sand and dirt) is nevertheless one of the important constituents, from both a purification and fertilizer point of view. Several of the larger scouring plants have tried methods of recovery based especially upon the content of potash and nitrogen obtainable in the finished product. In considering the possibility of utilizing waste scouring liquors in fertilizers, the forms in which the nitrogen existed in such liquors were determined. Practically all the nitrogen was present in the forms commonly found in the urine of animals and in the percentages shown in Table 4. This summary, although not complete, furnishes fairly comprehensive information on the more important compounds of nitrogen present in a representative sample of waste scouring liquors and their fertilizer value.

TABLE 4

PARTITION OF NITROGEN OF WASTE WOOL-SCOURING LIQUORS

	% of Total Nitrogen
As ammonia	2.4
As urea	18.0
As hippuric acid	11.6
As allantoin	10.9
As uric acid	11.8
As protein	28.8
As amino acids	3.1
As undetermined	13.4

Turning again to the handling of the waste liquors, it appeared that the most promising line of work would be a procedure which would give a waste liquor containing enough potash salts and nitrogenous matter to make, when evaporated, a fertilizer, recover a neutral wool grease, settle out dirt usable as a fertilizer filler, and yield water for use in boilers and for scouring. We realize, of course, that such a process would necessitate fundamental changes in the wool-scouring process commonly employed, which, we are frank to say, seems neither impracticable nor even undesirable. The demonstration on a commercial scale and the success of the proposed process will depend, of course, on the courageous support of a scourer or group of scourers, who have the means and the patience to work it out practically.

The process which we recommend as most promising, but for which no great claims of originality are made, consists in (1) extracting the grease with naphtha in keirs, as is now done in several large mills, then scouring with soap and water as usual; or (2) as the counter current system, scouring as usual with alkali and soap. In both cases the wash liquor is used until that in the first bowl is sufficiently concentrated to permit economical evaporation, or to about 20 to 25 per cent of total solids, including sand and dirt. In (1) the grease is recovered by distilling off the naphtha under reduced pressure and below 95 deg. Cent. In (2) the grease is removed with the centrifuge. The first-bowl waste liquor is further concentrated in triple or quadruple effect evaporators to 60 per cent solids and finally dried on a drum to a product containing approximately 5 per cent moisture.

In studying the proposed procedure samples of Australian wools when raw and after they had passed through the second bowl were obtained from several scouring plants. The composition of the matters removable from these wools is shown in Table 5. The results are based on the air-dry wool in the condition as received.

The quantities of wool scoured were as follows: No. 1, 11,973 pounds; No. 2, 10,881 pounds; No. 3, 11,764 pounds; No. 4, 11,800 pounds; No. 5, 10,146 pounds. All the wool was taken from a shipment of Australian 70's. Bowl No. 1 held 800 gallons, and No. 2, 1,000 gallons. "Red-oil potash" soaps were used in the scouring; therefore the liquors are a trifle higher in potash than they would be if sodium soaps had been used.

The figures in Tables 5 and 6 are regrettably inconclusive, as during the washing of each lot of wool both the first bowl and the second bowl were dumped four times each. The figures are encouraging, however, in showing that the greater part of each constituent is removed in the first two bowls, and also because laboratory tests indicate that these concentrations may be approached in scouring.

TABLE 5

MATTER REMOVABLE FROM WOOLS IN FIRST AND SECOND BOWLS

Sample	Composition						
	Moisture	Grease	Total Ash	Water Extract	Ash of Water Extract	K_2O in Water Extract	Nitrogen in Water Extract
No. 1:							
Raw	6.30%	17.01%	17.80%	15.81%	6.26%	3.47%	0.88%
End 2d bowl	7.28	3.17	3.06	2.13	0.62	0.23	0.12
No. 2:							
Raw	7.06	16.80	17.70	7.57	4.22	2.41	0.40
End 2d bowl	6.98	2.85	2.76	2.34	0.67	0.25	0.15
No. 3:							
Raw	7.96	16.06	16.85	11.08	5.31	2.82	0.40
End 2d bowl	6.74	4.34	3.59	2.84	0.84	0.44	0.18
No. 4:							
Raw	7.85	17.76	17.74	11.82	5.35	2.50	0.47
End 2d bowl	6.62	4.77	4.06	3.01	0.96	0.32	0.18
No. 5:							
Raw	7.76	20.33	12.96	10.03	4.52	2.42	0.43
End 2d bowl	7.81	1.59	3.05	3.62	1.26	0.23	0.16

TABLE 6

CONSTITUENTS REMOVED FROM RAW WOOL IN FIRST TWO BOWLS[1]

Sample	Constituents				
	Grease	K_2O	N	H_2O Extract	Ash
No. 1	11.0%	2.6%	0.5%	19.3%	11.5%
No. 2	10.0	1.6	0.2	3.6	10.9
No. 3	15.0	1.9	0.2	6.3	10.0
No. 4	10.2	1.7	0.2	7.3	10.2
No. 5	12.5	1.5	0.2	4.0	6.8

[1] These figures are calculated from the loss from the wools and are stated on the basis of the waste liquors in order to show what would have been the concentration of the waste liquors had it been practicable to scour all the wool of each lot without dumping the bowls.

Within recent years a large scouring plant in New England has conducted extensive large-scale experiments on concentrating its normal waste liquors from the first two bowls. This mill scours only wools of half-blood or higher grades and uses a red-oil potash soap as a scouring agent. As would be expected, its waste liquors were comparatively high in grease and potash.

TABLE 7

COMPOSITION OF WASTE-SCOURING LIQUORS FROM FIRST AND SECOND BOWLS

(*Liquors from Wools in Table 5*)

Sample	Bowl	Total Solids	Pet. Ether Ext'ct	Total Ash	K_2O	Nitrogen	Total Solids in Filtrate	K_2O in Filtrate
No. 1	1	5.1%	2.2%	1.3%	0.4%	0.12%	2.0%	0.5%
	2	7.5	3.6	1.9	0.7	0.17	2.6	0.7
No. 2	1	6.0	2.9	1.5	0.5	0.14	2.3	0.5
	2	7.3	3.3	1.8	0.6	0.16	2.6	0.6
No. 3	1	11.0	4.2	3.0	0.9	0.23	3.8	1.0
	2	1.7	0.9	0.4	0.1	0.02	0.8	0.1
No. 4	1	10.6	4.7	2.7	0.9	0.22	3.1	0.9
	2	1.8	0.9	0.4	0.1	0.03	0.7	0.1
No. 5	1	9.9	5.2	2.6	0.9	0.21	3.7	0.9
	2	1.7	1.9	0.4	0.1	0.03	0.8	0.1

Of all the normal first-bowl liquors examined during this work those listed in Table 7 are the most concentrated. The method employed at the mill in handling these liquors was as follows: As they were removed from the first and second bowls the waste-scouring liquors were conducted first into a settling tank, where a large part of the dirt, sand, and suspended matter settled. The partially purified liquors were then heated anh passed through a series of Sharpless centrifuges, which removed approximately 75 per cent of the grease, and then conducted into triple-effect evaporators, from which they emerged as a thick, brown-black, semi-solid mass, containing approximately 60 per cent of total solids, having the following composition: Total fatty matter, 14.2 per cent; volatile fatty acid as acetic, 6.1 per cent; grease soluble in petroleum spirit, 7.2 per cent; nitrogen, 1.2 per cent; ash, 21.7 per cent; moisture, 41.6 per cent; total potash (K_2O), 14.4 per cent; and water-soluble potash (K_2O), 12.7 per cent.

By further drying on a drum to 5 per cent moisture, the product would contain 22 per cent of grease, 1.97 per cent of nitrogen, and 20 per cent of water-soluble potash. The percentage of grease, although high, is not sufficiently high to make the product unsuitable as a fertilizer raw material. The value of the products obtained in this large-scale experiment on evaporating ordinary waste wool-scouring liquors, we are advised, slightly exceeded half the cost of the operations. We are not prepared to say, however, that the complete recovery and utilization of the wastes, the production of a pure soft water for boiler and scouring purposes, and the total elimination of all stream pollution, with the consequent sanitary and industrial advantages, may not justify this expense.

Laboratory Experiments to Obtain a Concerted Waste Liquor

Additional data on the practicability of obtaining a more concentrated liquor for evaporation and thus greatly reducing the cost of this operation were needed. Accordingly the Bureau of Chemistry undertook a series of small-scale washing experiments, using a naphtha-degreased Vermont low-fines wool having the composition shown in Table 8.

TABLE 8
Composition of Vermont Low-Fines Wool

	On Raw Wool	Degreased Wool
Moisture	2.6%	7.1%
Grease	16.1	—
Ash	15.3	13.2
Potash (K_2O)	4.8	5.0

Three pounds of the degreased wool was divided into six half-pound lots. These samples were placed in clean muslin bags. One bag was submerged in two pounds (935 c.c.) of warm water in a jar and agitated by hand for two minutes. The bag was then removed, squeezed as dry as possible, and placed in a second jar containing 935 c.c. of warm water (45 deg. Cent.). After agitating for two minutes, the bag was removed, squeezed, and placed in a third jar, and so on through eight jars. The solution removed from the jars by the sample was replaced in each jar by enough liquor from the succeeding jar (counter current system), the last jar being replenished with clean warm water. The other lots were then put through the same treatment as the first and so on until the six half-pound lots had passed through the solutions, which were then analyzed with the results shown in Table 9.

TABLE 9
Composition of Liquors from Degreased Wool

Jar No.	Total Solids	Soluble Solids	Grease	Soluble Potash (K_2O)	Nitrogen
1	22.9%	9.7%	1.3%	3.5%	0.8%
2	14.7	8.1	0.9	2.6	0.6
3	8.6	6.6	0.5	1.4	0.4
4	4.4	2.3	0.3	0.6	0.2
5	2.5	1.2	0.2	0.4	0.1
6	1.5	0.6	0.1	0.2	0.07
7	0.8	0.4	—	0.1	0.04
8	0.5	0.2	—	0.1	0.03

The volume and specific gravity of the liquid in each jar was as follows: No. 1, 500 c.c., 1.047; No. 2, 920 c.c., 1.059; No. 3, 1,020 c.c., 1.027; No. 4, 910 c.c., 1.010; No. 5, 910 c.c., 1.001; No. 6, 1,020 c.c., 0.98; No. 7, 930 c.c., 0.95; No. 8, 980 c.c., 0.93.

The liquors in jars 1 and 2 were rather thick and heavy, owing largely to the tendency of the albuminous matter to thicken slowly, even at 45 deg. Cent., and to the presence of a large quantity of sand and dirt, approximately 12 per cent in jar 1 and 6 per cent in jar 2. These liquors remained sufficiently fluid for washing the wool during the hour that was required to conduct the experimental washings at 45 deg. Cent., but set to a jelly on cooling overnight. It is doubtful, therefore, whether sedimentation of the liquor from the first bowl would be sufficiently rapid to materially reduce the proportion of suspended matter carried by liquor of this concentration. It seems probable, however, that if the liquor flowing form the second bowl to the first were passed through a centrifuge and the liquor withdrawn from the bottom of the front end of the first bowl, passed through a second centrifuge, and returned to the back end, a large part of the suspended matter could be removed, thus decreasing the cost of further concentration. However, experimentation on a large scale will be necessary to determine the feasibility of doing this.

The concentration of the liquor in the fourth jar is about the same as that of the ordinary soap and water "scour" in the first bowl (except, of course, in grease), and that of the liquor in the eighth jar approximates that of the liquor in the fourth bowl of the ordinary scouring liquors.

The results thus obtained plainly show that it is

possible to obtain a higher concentration of potash by using degreased wool; that more of such wool can be scoured in the same volume of water than when using raw wool, and that the potash concentration was not as high as expected, but that this concentration (3.5 per cent) could be treated, it is believed, on a commercial scale for the recovery of potash at a profit.

Fertilizer Materials from Waste Liquor

While the experimental work here outlined was in progress, experiments on working the waste into fertilizers were being conducted.

Sample No. 1, the concentrated waste liquor from New England, containing 60 per cent solids, 14.4 per cent potash, and 1.2 per cent nitrogen, was mixed with several phosphatic materials in several proportions, dried, and run through a small mill. Sample No. 2, made with 83 per cent acid phosphate and 17 per cent concentrated waste liquor, contains 0.3 per cent nitrogen, 15½ per cent phosphoric acid, and 2.2 per cent potash. Sample No. 3, made with 68 per cent acid phosphate and 32 per cent concentrated waste liquor, contains ½ per cent N, 14 per cent phosphoric acid, and 4.1 per cent potash. Sample No. 4, made with 46 per cent acid phosphate, 17 per cent raw bone meal, and 17 per cent concentrated waste liquor, contains 1.8 per cent N, 21 per cent phosphoric acid, and 2.3 per cent potash.

Two factory-scale tests were also run. In the first, in co-operation with J. C. Cottmann & Co., Baltimore, Md., 12 barrels (600 gallons weighing about 5,500 pounds) of the concentrated waste liquor was mixed with approximately the same weight (dry basis) of raw scrap leather. The mixture was steamed, dried and ground. Sample No. 3, which contains 6.1 per cent nitrogen and 6½ per cent potash, is representative of this product. In co-operation with the Standard Guano Company, Baltimore, Md., 100 barrels of the concentrated waste liquors were used in making a complete fertilizer for which the mixing formula was 810 pounds 54 deg. acid, 800 pounds ground phosphate rock, 550 pounds hair and wool waste, and ¾ barrel concentrated waste liquor. When the "den" was opened, 10 barrels of dark thin grease were collected. Sample No. 6 was taken from this material, which contains 0.95 per cent nitrogen, 15.17 per cent available phosphoric acid, 0.9 per cent potash, and 1 per cent grease. This in our experiments showed good results in growing wheat, corn, and cowpeas on soil of three widely different types.

Both of the factory scale experiments conducted to determine the practicability of working up the concentrated waste liquor proved entirely feasible and economical. The primary question with the fertilizer manufacturers is whether the material can be had in sufficient quantity and at a price per unit of nitrogen and of potash that will compare favorably with the prices of other raw materials. It is, of course, perfectly obvious that the initiative must come from the sources. They must furnish the material in quantities and at a price that will prove attractive to fertilizer manufacturers. We believe this is possible.

Value of Recoverable By-Products

It has been said that "Figures do not lie, but liars figure." By using high values and a good measure of optimism, large profits may often be shown upon paper—but never realized. We want to avoid such an outcome. We prefer to paint the picture darker than we believe it actually is.

From the average analytical figures obtained in examining the 300 samples of raw wool (slightly reduced) and the minimum market price of the several important constituents, it is practicable to obtain what we believe are reliable approxiate gross values for the by-products from any given quality of wool.

The estimated approximate value of wool waste by-products based on 100,000 pounds of wool is as follows:

Neutral grease (10 per cent): 10,000 pounds at 7 cents per pound	$700.00
Potash (K_2O) (3½ per cent): 3,500 pounds at 3 cents per pound[2]	105.00
Nitrogen (1½ per cent): 500 pounds at 15 cents per pound[3]	75.00
Filler (sand and dirt) (15 per cent): 15,000 pounds at 0.1 cent per pound	15.00
Total (29,000 pounds)	$895.00

[2] Basis of kainit price.
[3] Basis of garbage tankage price.

That is, a low estimate of yields from 100,000 pounds of average raw wool is approximately 10,000 pounds of neutral grease, worth about $700, and 19,000 pounds of fertilizer material, worth about $195, giving a total gross value of about $895.

Practicability of Waste Recovery and Utilization

This briefly covers our study of the problem. Our funds were exhausted at this stage where small industrial scale work is necessary in order to definitely answer the question, "Can waste wool-scouring liquors be economically recovered?" Like others who have studied the subject, we cannot answer the question categorically. Our firm belief, based on a careful consideration of the work of others and on our own experiments, is that it can be done. Perhaps it will be done at a considerable profit. Let us hope so. Perhaps it will be done at a return that barely covers the cost. Even so it is well worth the doing for sanitary and industrial reasons alone, and the present is as good a time as any to begin.

It should be undertaken and financed co-operatively by a group of wool scourers, at one of the scouring

mills, utilizing the experience which has been acquired during the past ten years.

It is going to require patience, courage, ability and money. If it were an easy problem, it would have been solved years ago. It may take one year and cost $25,000 or it may take five years and cost $200,000. It's worth it.

It is going to require modifications in present scouring machinery and perhaps profound changes in scouring processes. Here is where patience and courage are required, but there seems to be plenty of room for improvement.

If such a group, such funds, and such co-operation can be had, the Bureau of Chemistry will be glad to lend its earnest aid in solving the problems of the recovery and utilization of wool-scouring waste, in a united endeavor to improve sanitary and industrial conditions and to increase the fertilizer resources of the country.

Control of Hydrosulphite Vats

The Variables in the Process—Titration of the Titanium Chloride Solution—Controlling Conditions—Method Employed—Details of the Method—The Titration—Application of the Process—Concentration of the Liquor—A Quicker Method—Required Concentrations of Ingredients—The Time Concentration Curve—Example of Such a Process—Dyeing in Two Baths —Determining Least Allowable Concentration

By DR. ALEXANDER LAUTERBACH

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(*Translated by Ismar Ginsberg, B. Sc., Chem. Eng.*)

IN accordance with the opinion of the author the control of hydrosulphite vats depends on the determination of the temperature and of the specific gravity of the vat liquor and on its examination for color and clarity as far as outward appearance is concerned. A further test is the rapidity with which the dyed material turns green after the dye liquor is allowed to run away from it. This is also known as the glass plate test. As far as the alkalinity of the vat liquor is concerned, reliance is placed almost entirely on the feel of the liquor or of the goods by simply rubbing them between the fingers. Then again, frequently a short coloring test, lasting for a few minutes and consisting of a spot test on a piece of material, is employed to determine further properties of the dye liquor.

But aside from the determination of temperature and density of the liquor, all these tests yield qualtitative results only, for when quantitative results are required, these tests fail. It is for this reason that the author set to work to develop a comprehensive method for testing hydrosulphite vat liquor. The author has used his method in practical work for quite a long period of time, and he has obtained very good results with it. It has occurred to him that others might be interested in the matter as well.

The Variable in the Process

The most important variables in the dyeing process, namely temperature, concentration of the dye liquor and of the hydrosulphite in it, when determined quantitatively, will give a very accurate and detailed description of the composition of the dye bath in the form of a color diagram which can be used with advantage in a great many cases.

The amount of hydrosulphite that is present in the dye liquor was determined by the method of Knecht and Hibbert, which consists in the use of an aqueous solution of Methylene Blue. This solution is made by dissolving 10 grams of the dyestuff in 1 liter of water. In addition a solution of titanium chloride is used. The latter solution is made by digesting 50 c.c. of commercial titanium chloride with an equal volume of concentrated hydrochloric acid. The digestion lasts for a few minutes and the solution is then made up to the volume of 1 liter with the aid of air-free water. The solutions are placed in large flasks from which they are led into burettes. The Methylene Blue solution is kept in an atmosphere of carbon dioxide and the titanium chloride solution is maintained in an atmosphere of hydrogen gas.

Titration of the Titanium Chloride Solution

The titanium chloride solution is titrated with Mohr salt. Fourteen grams of this salt are dissolved in dilute sulphuric acid, and then the solution is made up to a liter volume. Fifty c.c. of this solution, containing one-tenth of a gram of ice, are mixed with a little fiftieth normal potassium permanganate solution for the purpose of oxidizing the ferrous iron to a light rose color. Then an excess of potassium sulphocyanide is added as an indicator and the solution is titrated with the titanium chloride. The strength of the Methylene Blue solution is determined by measuring off a definite quantity of the solution, and mixing it with acetic acid. This is titrated in a current of carbon dioxide gas with titanium chloride until the color is removed.

(The author discusses the difficulty of securing a proper endpoint in this titration and describes how he got around this difficulty by the use of a few tenths of a gram of salicylic acid. Salicylic acid seems to be possessed of catalytic powers in this connection.—TRANS.)

The catalytic action of salicylic acid is seen from the following experiment. A solution of safranin is mixed with an excess of titanium chloride solution which has been made strongly acid with hydrochloric acid. This is done at room temperature. When a small amount of salicylic acid is added thereafter, the safranin is almost immediately decolored.

Small quantities of sulphite and thiosulphite have no effect on the titration according to the results obtained by Knecht and Hibbert. (Various figures are given to indicate the truth of this statement.—TRANS.)

As far as the conditions that apply in skein and jigger dyeing processes are concerned, only small amounts of these materials are present so that they do not have any bad effect on the test. The concentration of coloring matter is small in most baths and hence the presence of the color in the dye liquor does not affect the recognition of the endpoint of the titration; on the other hand, the action that is exerted by the dyestuff on the reactions that take place in the titration is so small that it need not be taken into consideration.

CONTROLLING CONDITIONS

One essential condition which controls the obtaining of good results by this method is the absence of all alkali acetates. If they are present the addition of titanium chloride solution produces turbidity in the mixture which makes it practically impossible to tell the endpoint of the titration. Furthermore, the acetic acid added must be free of dissolved oxygen.

It is, however, possible to counteract the bad effect of alkali acetates by the addition of a little hydrochloric acid or sulphuric acid before the back-titration. Then again large amounts of mineral acids in the mixture are harmful when sulphites or thiosulphites are present. The removal of air from acetic acid is best accomplished by the addition to it of a little sodium bicarbonate.

METHOD EMPLOYED

The author obtained good results according to the following method: About 5 c.c. of the Methylene Blue solution in excess of that necessary to oxidize the hydrosulphite are allowed to flow into a titrating flask and then 5 c.c. of technical acetic acid are added (7 deg. Be.). Then a stream of carbon dioxide gas is passed through the apparatus and this is continued until the close of the test. About half a gram of sodium bicarbonate is added and 10 c.c. of the dye liquor are drawn off by a pipette and are introduced into the flask. This is first mixed with 40 c.c. of technical acetic acid to which half a cubic centimeter of concentrated hydrochloric acid and about half a gram of sodium bicarbonate have been added. Then about 0.15 gram of salicylic acid is added and the titration is carried out by means of a standard solution of titanium chloride.

The color change that takes place in the solution is from bright blue and green, according to the concentration of the dye liquor, to a yellow and brown. If a dirty blue green is obtained it is an indication that the endpoint of the titration has been overstepped. This is very apt to happen when the solution becomes cloudy, which is due to an insufficient amount of mineral acid being present in it. This is easily determined by the addition of a little hydrochloric acid which will instantly cause the solution to clear up. But when the cloudiness of the solution is due to the presence of sodium thiosulphate, then the addition of hydrochloric acid will not make it disappear. In this case it is impossible to prevent too high results from being obtained from the analysis It must also be mentioned that the presence of large amounts of zinc chloride also has a bad effect on the color change.

The addition of sodium bicarbonate to the solution is of considerable importance, and in the original article the author gives a number of figures to show what exact effect this salt has on the titration.

It is advisable to keep the solution of titanium chloride in an atmosphere of carbon dioxide gas, and the solution is also covered, both in the large storage bottle as well as in the burette, with a film of paraffine oil. When the solution is protected in this manner, its lasting qualities are good. For example, the activity of such a solution was found to decrease from 6.47 to 6.70 c.c. in terms of 10 c.c. of the Mythylene Blue solution. In the analytical process, which has just been described, great care must be taken to avoid any trace of aldehyde, for this has a very serious effect on the accuracy of the method.

HOW THE METHOD IS EMPLOYED

When the hydrosulphite vat contains a high concentration of coloring matter this method cannot be applied directly to the dye liquor. The same condition holds true for the liquor from the continuous vat. As an example of such liquor the Indigo hydrosulphite vat is taken.

The first disturbing action is caused by the too high concentration of indigo white in the liquor. In the first place this substance is oxidized by Methylene Blue and then again it renders it practically impossible to detect the endpoint of the titration. Furthermore, when the dye bath has been used for a long time, it contains such large quantities of sodium sulphite that the disturbing action of this substance on the titration can no longer be neglected.

Thus, in order to apply this analytical process to the vat liquor, it is first necessary to remove the indigo white and to render both the sulphite and the thiosulphite harmless. But this must be done in such a manner that the hydrosulphite in solution is not harmed. Formaldehyde is used for stabilizing the hydrosulphite. When a sample of the vat liquor is permitted to flow into the

formaldehyde bath, then the indigo white may be precipitated by the addition of either acids or salts. The addition of the vat liquor to acetic acid seems to be undesirable from two standpoints. In the first place, the sodium hydrosulphite decomposes very rapidly in acetic acid solution, and in the second place a neutral solution is required after filtering at a latter stage of the process. When acetic acid is added, this cannot be effected without additional effort.

This disadvantage is counteracted by precipitating the indigo white by means of a solution of sulphate of zinc. It is advisable to add a little acetic acid to this solution. Therewith a great amount of carbon dioxide gas is evolved when sodium bicarbonate is added for the purpose of removing the air in the solution.

The precipitation is carried out in a measuring flask which is filled to the mark with formaldehyde and which is allowed to remain for a few minutes to ensure complete formation of formaldehyde sulphoxylate and formaldehyde bisulphite as well as for stabilizing the hydrosulphite. The precipitate of indigo white is removed and an aliquot portion of the filtrate is taken for analysis.

Exact Details of the Method

The exact details of the method are as follows:

Five c.c. of a 50 per cent solution of zinc sulphate are filled into a 100 c.c. measuring flask and a stream of carbon dioxide is passed through the bottle in such a manner that the inlet tube is not permitted to dip into the solution. The inlet tube can be easily moved so that its position with respect to the level of liquor may be changed at will. When all the air has been removed from the bottle, about half a gram of sodium bicarbonate is added. A solution of formaldehyde and ammonium carbonate is made in another flask or beaker. About 80 grams of formaldehyde and half to 1 gram of the carbonate of ammonia, pulverized, are used. Evolution of gas takes place.

Then the end of the carbon dioxide inlet tube is raised above the opening of the measuring flask and 20 c.c. of the vat liquor are allowed to flow into the flask from a pipette which has two marks on it. The end of the pipette is placed as close as possible to the level of the liquid in the flask, and the carbon dioxide inlet tube is pushed into the neck of the bottle.

The formaldehyde which has been mixed with the ammonium carbonate, and which still effervesces, is filled into the measuring flask while raising the carbon dioxide inlet tube until the level of the liquid is at two-thirds of the volume of the flask. The flask is then shaken and finally filled to the mark. Then the inlet tube is raised out of the flask and the mouth is corked with a stopper. The flask is thoroughly shaken so that the contents of the same are well mixed and thereafter it is allowed to stand for five minutes. There is no error introduced in the analysis when the flask is allowed to remain standing for considerable periods of time, as much as two to three hours.

The Titration

A solution of Methylene Blue is filled into a titrating flask and 5 c.c. in excess of the amount required are added. This solution is mixed with 5 c.c. of technical acetic acid. A thermometer is placed in the flask and the carbon dioxide inlet tube is arranged so that its end reaches below the level of the liquid. Then sufficient distilled water is added so that the bulb of the thermometer is surrounded with it. The flask is then placed on a tripod.

The vat liquor solution, mentioned above and allowed to stand for five minutes, is then filtered in the ordinary manner into a dry breaker and at the same time the titration flask is heated, after the stream of carbon dioxide gas has been allowed to flow through it for a moment or two at the rate of about thirty bubbles of the gas in a second, and after a small amount of sodium bicarbonate has been added. The heating progresses until the temperature reaches 90 deg. Cent. When this temperature is attained and without interrupting the heating process, 50 c.c. of the filtrate are permitted to flow into the titrating flask and the heating is continued. Then again, 40 c.c. of technical acetic acid are added, which has a half a cubic centimeter of concentrated hydrochloric acid. A little sodium bicarbonate is added as well.

When the temperature of the contents of the flask reaches 90 deg. Cent., which takes place after three to five minutes, the flask is removed from the flame. The 40 c.c. of acetic acid are added, about 0.15 gram of salicylic acid is introduced and the solution is titrated with a standard solution of titanium chloride. The stream of carbon dioxide gas must be kept flowing through the apparatus drying all this time.

Application of the Process

This method is applicable to the analysis of all sorts of hydrosulphite vat preparations, containing various dyestuffs, which can be precipitated in the manner which has just been described. The method is also suitable for the determination of the sulphoxylate content in Rongalite C and CL. Leucotrope does not introduce any disturbing factors. The method is also applicable to the determination of the sulphoxylate present in printing pastes. In this case, glue gelatine, blanc fixe, starch pastes, zinc oxide, Turkey Red oil and leucotrope do not disturb the process, particularly in the quantities in which they exist in printing pastes of the ordinary sort used in white discharge processes.

The analysis of solid rongalite and printing pastes is carried out in the same manner. A measured sample of the substance is placed in a titrating flask and then mixed with Methylene Blue, 5 c.c. of acetic acid and a little sodium bicarbonate. The mass is heated in a current of carbon dioxide gas to 94 deg. Cent., and is then mixed with 40 c.c. of acetic acid and the titration proceeds then in the same manner as described above.

Very accurate results are obtained by this method of analysis. It appears to be a complicated method, but it

gives the information that the dyer wants to know about the hydrosulphite vat and which he can use to good advantage in controlling the dyeing operation. An analysis takes twenty minutes. The method has been worked out so that it is eminently fitted for technical purposes. But then again it yields absolutely accurate results which is an additional advantage. It thus forms a very valuable addition to the technical information available on the control of hydrosulphite vat liquors.

Concentration of the Liquor

The determination of the concentration of the lye in the vat has been a difficult matter because of the interference offered by both the hydrosulphite and the dyestuffs. Furthermore, it must be remembered that a part of the alkali is present in the liquor as carbonate and not as hydroxide. Then again, the sodium which may be combined with the dyestuff is always determined as free caustic soda.

The following method is a useful one in this connection: The sample of vat liquor is permitted to flow into a measuring flask. A definite amount of a solution of barium chloride and sufficient hydrogen peroxide are placed in the flask before allowing the dye liquor to run into it. Then the flask is filled to the mark and the contents are filtered through a dry filter; the caustic soda in the solution is determined by taking an aliquot part and titrating with a standard solution of hydrochloric acid, using Methyl Orange as an indicator.

The alkalinity of the carbonate and the sulphite present in the solution are counteracted by precipitation with barium chloride. The object of the hydrogen peroxide is to oxidize the hydrosulphite and the reduced dyestuff. The product obtained from the hydrosulphite is principally bisulphate, whereas 2 molecules of caustic soda are neutralized by 1 molecule of the hydrosulphite. Hence the results obtained from the titration will be short a certain quantity of caustic soda, which is equivalent to 2 molecules of the alkali for every one molecule of hydrosulphite present in the vat liquor. That means that 0.46 grams of NaCH is equivalent to 1 gram of hydrosulphite and to 1.54 c c. of a 30 deg. Be. solution of sodium hydroxide. A proper correction must thus be made.

A Quicker Method

In many cases the accuracy of the analysis is satisfactory if it is equivalent to 1 to 2 c.c. of a 30 deg. Be. solution of sodium hydroxide. Hence, a quicker method may be used as explained below.

A solution of caustic soda is employed as the titrating solution. This solution of caustic contains 10 c.c. of 30 deg. Be. solution of sodium hydroxide in each liter. Another solution is necessary, that of an acid, and for this purpose a dilute solution of acetic acid is employed. This solution of acetic acid has been proven to have the same strength, that is, the same normality, as the caustic soda solution. The test is made with phenolphthalein. A 10 c.c. sample of the vat liquor is employed and the number of cubic centimeters of the acetic acid solution required for neutralization gives the content of caustic soda in the vat liquor in terms of 30 deg. Be. alkali per liter.

When the concentration of the alkali on the dye liquor is small or when the color of the dyestuff prevents the sharp definition of the color change, it is advisable to allow an excess of acetic acid solution to run into the sample of hydrosulphite vat liquor and the excess acid is titrated back with the solution of alkali until a red color forms in the latter. If the dyestuff exerts a disturbing action on this analysis as well, then the solution is made up to a definite volume, before being titrated with the sodium hydroxide solution; it is filtered through a dry filter and the excess of acid is determined in an aliquot portion of the filtrate.

The results obtained in this manner on the concentration of alkali and hydrosulphite in the vat liquor are extremely useful in controlling the dyeing process which is carried out with this liquor. But in order to make use of this information, it is also necessary to know each and every factor that controls the quantities of these materials that must be employed so that the exact quantities of the lye and of the hydrosulphite, required to give best results in the vat dyeing, may be accurately known.

Required Concentrations of Ingredients

The usual formulae for making up the vat liquors do not give very much information, or any at all, on this subject. They just give in more or less details mere instructions as the manner in which the color bath should be prepared. When the directions are closely followed, the composition of the bath will be in general what it was intended to be, but nevertheless there will be no exact information available as to its exact composition. Naturally little will be known in these circumstances of any variation that takes place in the various important factors that control the dyeing operation.

Then again, if consideration is given to the fact that in the dye plant there may be working operators possessed of greater or lesser skill in following out directions relating to the preparation of color baths, who may use more or less care in this important stage of the dyeing process, it follows that there is still more chance for variations to occur in the composition of the dye liquor with accompanying changes in the mode of dyeing. These changes will be recognized too late, when the dyed goods are seen to be faulty. Perhaps one of the most difficult tasks, then, is to trace back the fault to its proper source so as to eliminate any chance of its occurring again. Then again, mention must be made at this point of the variations that are inevitably introduced in carrying out on a technical and commercial scale the results that have been obtained in laboratory experimentation and that have been put out in the form of a formula for preparing the dye liquor.

The Time Concentration Curve

Errors that are made in this manner can only be detected and corrections applied to prevent their reoccurrence when the time-concentration curve for alkali and hydrosulphite has been plotted. When this curve is prepared with the time-temperature curve and the time-density curve, then a real picture of the entire dyeing process is available. This information describes the process in all its details, with the exception of the dyestuff, and forms the basis for a true control of vat dyeing.

Example of Such a Process

The production of a deep green color is an example of such a color picture. This green was made from a mixture of 0.6 per cent of Indanthrene Blue RS powder, 0.625 per cent of Indanthrene Golden Orange G powder and 12 per cent of Anthraflavone Paste. The time elements were plotted on the X axis and each division was equal to five minutes. On the ordinate axis the different variables were plotted, and each division was equal to the following:

1. In the case of temperature curve, 10 deg. Cent.
2. In the case of the liquor curve, 1 c.c. of 30 deg Be. caustic lye.
3. In the case of the hydrosulphite curves, 0.1 gram of hydrosulphite.
4. In the case of the density curve, 0.02 (in this case the zero point is 1 and not zero).

In the first experiments the dyeing was carried out with Indanthrene Blue RS, Indanthrene Golden Orange and Indanthrene Yellow G. But because of the trouble encountered with Indanthrene Yellow, which was not fast but ran when the cloth was cooked with soap solution and soda, another dyestuff had to be substituted. Anthraflavone was used instead. But as this dyestuff is not absorbed well by the fiber, the dyeing had to be carried out in two stages. This was done in the following manner:

Dyeing in Two Baths

Indanthrene RS and Indanthrene Golden Orange G were mixed with 20 c.c. of lye and the dyeing was continued for twenty minutes. Then it was interrupted and the alkalinity of the solution was reduced by the addition of acetic acid to 5 c.c. per liter. Then the Anthraflavone was dissolved in the dye liquor and the dyeing continued for another forty-five minutes. The advantages gained by this process are as follows:

Change of vats and the washing, centrifuging and other operations connected therewith are avoided. Hydrosulphite consumption is reduced, as the residue that remains from the first part of the process may be used in the second part. Furthermore, dyestuff consumption is reduced, as in the second process a redyeing takes place rather than any dyestuff being dissolved out of the cloth.

The curve diagrams drawn from the data furnished for this process indicated that the concentration of hydrosulphite in the dye liquor was much too great. Hence it was reduced to about half a gram per liter. A similar reduction of hydrosulphite concentration was possible in the second part of the process, although this reduction was not as large as in the first part.

It is understandable that a complete picture of the dyeing operation can be obtained by the plotting of the curves mentioned above, but this is generally not done unless certain difficulties arise in the process or when it is desired to economize on the use of reagents in the dyeing operation. In most cases it is sufficient to establish the values of the variables at the beginning of the dyeing. The decrease of concentration of hydrosulphite and also of the alkali in the dye liquor generally take place in the same manner for various color baths.

Then again, the titration of the hydrosulphite may be greatly simplified. In most cases the question that arises during the progress of the dyeing operation is whether sufficient hydrosulphite is present in the liquor or whether additional hydrosulphite need be added. It is well known just how high the concentration of hydrosulphite must be at a minimum in the dye liquor.

Determining the Least Allowable Concentration of Hydrosulphite

In order to determine whether the minimum quantity of hydrosulphite is present in the dye liquor, enough of a solution of Methylene Blue, equivalent to 10 c.c. of the vat liquor are placed in a test tube. A little acetic acid is added and 10 c.c. of dye liquor are allowed to flow into this mixture from a measuring pipette. If decoloration takes place then it may be concluded that there is sufficient hydrosulphite in the vat liquor. If decoloration does not take place then additional vat liquor is allowed to flow into the mixture until all of the Methylene Blue has been reduced. It is then possible to deduct from the quantity of vat liquor that has been required to effect decoloration of the Methylene Blue solution, whether the deficiency of hydrosulphite is large or small.

When determining any variable in the color process, it is essential to draw a picture of the effect which this variable has on the dyeing operation as a whole. It must be remembered that all the factors are intimately interconnected and that the individual factor has an effect on the following processes that take place during the dyeing operation; the reduction process, the solubility of the various materials used, the dyeing operation itself, that is, the absorption of dyestuff by the textile fiber, the nature of this absorption and the degree to which it takes place.

In general, the following remarks may be made regarding this problem. The temperature bears an important and direct relationship with the speed of the reduction of the dyestuff. Furthermore it also controls the degree to which reduction takes place. It affects the solubility of the reduced dyestuff and the absorption of the same by the fiber, the nature of this absorption and the degree

to which it occurs. It also affects the speed of the decomposition and oxidation of the hydrosulphite and therewith has a definite action in lowering the concentration of the lye. This action is direct and when the reduction in the concentration of alkali is corrected, it has the effect of increasing the specific gravity of the vat liquor. These are the effects of temperature variation.

The concentration of the hydrosulphite in the liquor limits and varies the speed of reduction and the degree of reduction of the dyestuff. It affects the coefficient of distribution of the coloring matter between the dye liquor and the fiber, from the standpoint of the speed with which the dyestuff goes on the textile fiber. When the hydrosulphite is oxidized hydrogen ions are formed and this phenomenon has the effects, of course, at reducing the alkalinity of the liquor.

The concentration of alkali in the dye liquor limits the solubility of the reduced dysetuff and accelerates the reduction of the same in this manner. However, the presence of sufficient alkali is not in all cases absolutely essential to the reduction of the dyestuff. The concentration of alkali also has an effect on the coefficient of distribution and on the speed with which the dyestuff is absorbed by the fiber. The specific gravity has some affect on the evenness of the dyeing or on the coefficients of distribution.

The concentration of coloring matter affects the concentration of alkali and of hydrosulphite among other factors as well.

This short description of the relationship that exists between the variables and the chemical and physical processes that take place during the dyeing operation must be added to by stating that the character of the dyestuff itself has an important bearing on the matter and can alter these inter-relations. It is not practical to determine what changes take place in this respect.

Dyeing Acetate Silk

Part IX

(Continued from page 424)

Special Developed Dyes for Acetate Silk—The Azoniles—Dyeing Acetate Silk with the Acedronoles—Formulas and Methods for Obtaining Two-Color Effects on Unions—Silkons and Azonines on Acetate Silk—Dyeing Acetate Silk with the Azoles

By CHAS. E. MULLIN, M. Sc., F. A. I. C.

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THE Azonile, Silkon, Acedronole, Azole, Azoic and certain Azonine dyes are developing dyes, usually amino and diamino compounds, which are applied to acetate silk, diazotized and developed. The following dyeing instructions will give many points on the application of these products, as well as other bases, etc., to acetate silk.

Dyeing Acetate Silk with Azoniles

Method No. 70: These products are applied to the scoured but unsaponified acetate silk. Some stain the acetate silk before developing whereas others leave it colorless, but on diazotizing and developing them on the fiber they yield a large number of shades ranging from yellow to black according to the developer used. The manufacturer only recommends *B*-naphthol, resorcinol and Dianil Developer O, but possibly phenol, *B*-hydroxynaphthoic acid, *A*-naphthol and other developers could also be used to give additional shades.

The Azoniles are prepared for use by mixing into a paste with the necessary quantity of concentrated hydrochloric acid (see Table No. IV), and adding 100 parts of boiling water to each part of dye and finally heating to the boiling if necessary. Actual boiling of the solution should be avoided in the case of Azonile R, which should simply be made into a paste with the acid, the water added at 90 deg. Cent. (195 deg. Fahr.) and allowed to stand for fifteen minutes at this temperature. Clear solutions will be obtained with Azoniles N and SR, which may be strained into the lukewarm dye bath direct. In a 1:100 dilution Azonile N tends to precipitate on cooling, therefore the solution should be very hot when adding it to the dye bath. Azoniles G, R and B leave some residue when dissolving; therefore particular care should be exercised to filter them.

TABLE IV

Amount of Hydrochloric Acid Used in Dissolving the Azoniles

Require the following number of parts of concentrated hydrochloric acid:

Each 100 parts of:	
Azonile G	70
Azonile N	60
Azonile R	100
Azonile B	60
Azonile SR	60

The goods are entered into a lukewarm 30 or 40 to 1 dye bath, working well and gradually adding during the dyeing from 10 to 20 per cent of ammonium acetate in solution to aid exhaustion. In the case of Azoniles G, R and B the ammonium acetate should be added very gradually in order to prevent precipitation of the Azonile. The temperature is raised during a half hour to 60 to 70 deg. Cent. (140 to 160 deg. Fahr.) and the dyeing continued for a half hour at this temperature, when the bath is usually exhausted. The goods are rinsed well before diazotizing.

Method No. 70A: The thoroughly rinsed goods are diazotized in a cold 1:20 bath for thirty minutes, containing 2 to 5 per cent of sodium nitrite and 4 to 10 per cent of concentrated hydrochloric acid, on the weight of the goods, depending upon the depth of shade. They are rinsed well and should be immediately developed.

Method No. 70B: The developers are dissolved in hot water, adding, in the case of *B*-naphthol, 70 parts of 77 deg. Tw. caustic soda solution, or in the case of Dianil Developer O, 30 parts of soda ash to every 100 parts of developer. The 1:20 developing bath is prepared, according to depth of shade, with 2 to 6 per cent of developer on the weight of the goods. The goods are entered cold and the bath slowly heated to 49 deg. Cent. (120 deg. Fahr.), working the goods for forty-five minutes. In case a standing bath is used for diazotizing, it should be replenished after each run with about one-third the original quantity of acid and nitrite. The same proportion of developer should be added to the developing bath if a standing bath is used. After developing the acetate silk is rinsed well, hydroextracted and dried at a low temperature, not over 49 deg. Cent. (120 deg. Fahr.). The brilliancy of the color is increased by a light soaping before drying.

The Azoniles give good full shades of excellent fastness to water, and with the exception of Azonile R which turns somewhat yellow to washing. Azonile G, even when not diazotized and developed has very good fastness properties, with the exception of Azonile R developed with *B*-naphthol and particularly Azonile B with Dianil Developer O, all of the Azoniles are very fast to light. Table No. V gives a few of the colors obtainable with these dyes. They may readily be used for two-color effects on unions of acetate silk with cotton or/and other artificial silks, by first applying the Azoniles as described above, which only dye cotton faintly, and afterward cross dyeing at 60 to 70 deg. Cent. (140 to 160 deg. Fahr.) in a salt bath containing no soap or alkalies, with direct cotton colors which do not appreciably stain acetate silk.

TABLE V

Formula No. and Color		*Dye and Developer*
Z1—Yellow	Azonil G.	Dev., none; dyed direct.
Z2—Scarlet	Azonil G.	Dev., B-naphthol.
Z3—Dull yellow	Azonil N.	Dev., Resorcinol.
Z4—Orange	Azonil R.	Dev., Resorcinol.
Z5—Cerise	Azonil R.	Dev., B-naphthol.
Z6—Violet	Azinol R.	Dev., Developer O.
Z7—Bright blue	Azinol B.	Dev., Developer O.
Z8—Red brown	Azinol SR.	Dev., Resorcinol.
Z9—Navy blue	Azinol SR.	Dev., Developer O.
Z10—Black	Azinol SR.	Dev., Developer O.

The following direct cotton dyes may be used as above and do not dye acetate silk: Oxydianil Yellow O, Dianil Direct Yellow S, Dianil Fast Orange 2R, Dianil Fast Scarlet 4BSN, Dianil Light Red 8BW Dianil Fast Blue GL, Dianil Blue B, G, R, H2G and H3G, Dianil Pure Blue PH, Dianil Fast Gray 2BL and Dianil Black ES. The following dyes do not stain acetate silk appreciably when applied in light shades: Aurophenine O, Dianil Yellow 3G, Dianil Fast Scarlet 8BS, Dianil Light Red 8BL, Dianil Pink BD and Dianil Fast Blue RL.

Dyeing Acetate Silk with Acedronoles

Method No. 71: In preparing the Acedronole dye bath, dissolve Acedronole DA, B and T in hot water without any addition. Acedronoles ND, CA, BN and AN are wet out with 50 to 100 times their weight of boiling water and dissolved by the addition of double their weight of concentrated hydrochloric acid. In other words, for Acedronoles ND, CA, BN and AN, 1 pound of the dye is mixed with 6 to 12 gallons of boiling water, and then 2 pounds of hydrochloric acid is added to complete the solution.

Acedronole AB is dissolved by mixing with ten times its weight of cold 85 per cent formic acid, about 100 times its weight of boiling water is added to this and then the remaining dye is brought into complete solution by the addition of four volumes of concentrated hydrochloric acid. Or in other words, 100 grams (about 3.5 ounces) of Acedronole AB should be mixed with a liter (slightly over a quart) of 85 per cent formic acid, about 10 liters (2.7 gallons) of boiling water is added to the formic acid-dye mixture and after stirring well, 400 c.c. (about 13.8 fluid ounces) of concentrated hydrochloric acid is added to complete the solution.

Acedronole AT is dissolved in exactly the same way as Acedronole AB, except that just twice the amount of hydrochloric acid given for AB is used. In other words, for 100 grams of AT, use 1 liter of formic acid, 10 liters of boiling water and 800 c.c. (about 27.5 5 fluid ounces) of concentrated hydrochloric acid. The dye bath is prepared by straining the above acedronole solution through cotton into the bath at about 40 deg. Cent. (104 deg. Fahr.).

The clean acetate silk is entered into about a 1 to 20 dye bath at 40 deg. Cent. (104 deg. Fahr.) and worked for about fifteen minutes. The temperature is raised to 60 to 70 deg. Cent. (140 to 158 deg. Fahr.) and the handling continued for about half an hour. Where required as given in the following formulas, the stated quantity of sodium acetate, in solution, is then added slowly to the bath to aid exhaustion, and after a further

working for about half an hour a rinse of cold water is given and the acetate silk is ready for diazotization.

Acedronoles ND, CA, BN, AN, DA, T and B may be mixed together in any proportions in the dye bath, and Acedronoles AB and AT may also be used together but should not be mixed with the above on account of the difference in the acidity of their baths, which may cause precipitation of the Acedronoles AB and AT in the less acid bath. Sodium acetate is not used with Acedronoles AB and AT.

Method No. 71A: The diazotizing bath for heavy shades is prepared cold with 4 per cent sodium nitrite and 10 per cent of concentrated hydrochloric acid, but for light shades a bath of half strength is sufficient. The acetate silk is treated cold, rinsed well and immediately developed. Also see Method No. 70A.

Method No. 71B: Phenol, resorcinol, *A*-naphthol, *B*-naphthol, Developed BON and Oxamine Developer B are used with the Acedronoles. The developing bath is prepared by dissolving phenol or resorcinol directly in hot water without any other addition. Oxamine Developer B is dissolved in hot water with the addition of a little hydrochloric acid. *B*-naphthol, *A*-naphthol and Developer BON are first made into a paste with hot water and an equal quantity of 38 to 40 deg. Be. caustic soda solution. This paste is dissolved by adding hot water. The rinsed acetate silk fresh from the diazotizing bath is entered in the cold developing bath, except in the case of Developer BON, and worked for about half an hour. In using Developer BON, 2 to 4 c.c. of 6 deg. Be. acetic acid per liter of bath must be added to the developing bath so that the bath will react acid to litmus paper when in use and Developer BON is used at 60 to 70 deg. Cent. (140 to 158 deg. Fahr.) instead of cold, as for the other developers.

After developing, rinse and scroop in the regular way. A warm soaping with a solution containing 2 to 3 grams per liter of Marseille soap, after developing and rinsing, as in Method No. 70B, will increase the luster of the acetate silk. In the following formulas cotton is left white or is only slightly stained, with the exception of formulas number X7, X8, X32, X33 and X43, so that all other formulas may be used for two-color effects on unions containing acetate silk and cotton or other artificial silks:

Formula No. and Color — *Base and Developer*

X1—Yellow:
1% Acedronole AB. Dev., 1.5% Phenol.

X2—Reddish Orange:
1% Acedronole AB. Dev., 1.5% Resorcinol.

X3—Orange Red:
1% Acedronole AB. Dev., 1.5% B-naphthol.

X4—Henna:
1% Acedronole AB. Dev., 1.5% A-naphthol.

X5—Cardinal:
0.7% Acedronole AB. Dev., 3% Developer BON.

X6—Dark Cardinal:
1% Acedronole AB. Dev., 1.5% Oxamine Developer B.

X7—Brown:
1% Acedronole ND with 5% sodium acetate. Dev., 1% Resorcinol.

Formula No. and Color — *Base and Developer*

X8—Darker Brown:
1.5% Acedronole DN with 6% Sodium Acetate. Dev., 1.5% Resorcinol.

X9—Orange Scarlet:
2% Acedronole CA with 6% Sodium Acetate. Dev., 1.5% Resorcinol.

X10—Bright Scarlet:
1% Acedronole CA with 4% Sodium Acetate. Dev., 2% Developer BON.

X11—Darker Scarlet:
1.5% Acedronole CA with 6% Sodium Acetate. Dev., 3% Developer BON.

X12—Orange:
1.25% Acedronole BN with 6% Sodium Acetate. Dev., 1.5% Resorcinol.

X13—Dark Orange:
1.25% Acedronole BN with 6% Sodium Acetate. Dev., 1.5% B-naphthol.

X14—Orange Brown:
1% Acedronole AN with 6% Sodium Acetate. Dev., 1.5% Phenol.

X15—Darker Than X14:
1.5% Acedronole AN with 8% Sodium Acetate. Dev., 1.5% Phenol.

X16—Brown, Reddish:
1% Acedronole AN with 6% Sodium Acetate. Dev., 1.5% A-naphthol.

X17—Darker Than X16:
1.5% Acedronole AN with 8% Sodium Acetate. Dev., 2% A-naphthol.

X18—Darker Than X17:
1% Acedronole AN with 6% Sodium Acetate. Dev., 1.5% Resorcinol.

X19—Darker Than X18:
1.5% Acedronole AN with 8% Sodium Acetate. Dev., 2% Resorcinol.

X20—Darker Than X19 (very dark):
1% Acedronole AN with 6% Sodium Acetate. Dev., 1.5% B-naphthol.

X21—Deep Blue:
1% Acedronole AN with 6% Sodium Acetate. Dev., 2.5% Developer BON.

X22—Black:
3% Acedronole AN with 10% Sodium Acetate. Dev., 5% Developer BON.

X23—Yellow:
1% Acedronole AT. Dev., 1.5% Phenol.

X24—Reddish Tan:
1% Acedronole AT. Dev., 1.5% Resorcinol.

X25—Bright Red:
1% Acedronole AT. Dev., 1.5% B-naphthol.

X26—Darker and Browner Than X4:
1% Acedronole AT. Dev., 1.5% A naphthol.

X27—Cerise:
1% Acedronole AT. Dev., 1.5% Oxamine Developer B.

X28—Dark Cerise:
0.7% Acedronole AT. Dev., 3% Developer BON.

X29—Medium Blue:
1% Acedronole DA 30% Paste with 2% Sodium Acetate. Dev., 2% Developer BON.

X30—Darker Than X29:
1.5% Acedronole DA 30% Paste with 4% Sodium Acetate. Dev., 2.5% Developer BON.

X31—Darker Than X21 or X30:
2% Acedronole DA 30% Paste with 6% Sodium Acetate. Dev., 3% Developer BON.

X32—Blue Black:
6% Acedronole DA 30% Paste with 10% Sodium Acetate. Dev., 5% Developer BON.

Formula No. and Color — *Base and Developer*

X33—Black:
6% Acedronole DA 30% Paste with 10% Sodium Acetate. Dev., 1% Phenol.

X34—Bright Light Yellow:
2% Acedronole DA 30% Paste with 4% Sodium Acetate Dev., 1.5% Phenol.

X35—Lighter Than X24:
2% Acedronole B 30% Paste with 4% Sodium Acetate. Dev., 1% Resorcinol.

X36—Red Henna:
4.5% Acedronole B 30% Paste with 6% Sodium Acetate. Dev., 1.5% Resorcinol.

X37—Darker Than X27:
6% Acedronole B 30% Paste with 6% Sodium Acetate. Dev., 2% B-naphthol.

X38—Lavender Blue:
2% Acedronole B 30% Paste with 4% Sodium Acetate. Dev., 2.5% Developer BON.

X39—Darker Than X38:
3% Acedronole B 30% Paste with 6% Sodium Acetate. Dev., 3% Developer BON.

X40—Slightly Darker Than X34:
2.5% Acedronole T 30% Paste with 5% Sodium Acetate. Dev., 1.5% Phenol.

X41—Dark Red:
4% Acedronole T 30% Paste with 6% Sodium Acetate. Dev., 2% B-naphthol.

X42—Red Brown or Dark Henna:
3.5% Acedronole T 30% Paste with 6% Sodium Acetate. Dev., 1.5% Resorcinol.

X43—Dark Tan:
1% Acedronole BN and 0.4% Acedronole ND with 6% Sodium Acetate. Dev., 1.5% Resorcinol.

X44—Darker Than X43:
0.5% Acedronole AN and 0.2% Acedronole BN with 3% Sodium Acetate. Dev., 1% Resorcinol.

These shades have a good fastness to water, washing, dilute organic acids, ironing, and, with the exception of formulas number X6, X12, X13, X37 and X41, a good fastness to light. They will stand acid cross-dyeing of wool, with the exception of formulas number X2, X3, X5, X9, X10, X11, X18, X19, X20, X24, X25, X28, X34, X35, X36 and X37. Where it is decided to dye the other fiber combined with the acetate silk, the acetate silk should be dyed first according to Methods No. 71, 71A and 71B, and the cotton then dyed with such substantive dyes as will not cover acetate silk, at about 40 to 60 deg. Cent. (104 to 140 deg. Fahr.), or at a maximum of 80 deg. Cent. (176 deg. Fahr.). The following dyes have proven suitable: Stilbene Yellow 3G and G, Pyramine Yellow G, Cotton Orange G and R, Pyramine Brilliant Orange 3RS, Thiazine Brown G and R, Oxamine Brilliant Red B, Oxamine Fast Pink B, Cotton Pink GN and BN, Thiazine Red G and R, Oxamine Blue GN, B, 3B and 4B, Oxamine Dark Blue BG, Oxamine Light Blue B, Oxamine Pure Blue 5B and 6B, Oxamine Black RN and BHN, and, for black, Oxamine Black RN or BHN, diazotized and developed with resorcinol.

Silkons on Acetate Silk

Very little information regarding the Silkons has been available, as they are at present possibly not offered in America. However, it is understood that Silkon B corresponds to Acedronole AB, while Silkons C and D correspond to Acedronoles CA and DA, respectively.

Developed Azonines on Acetate Silk*

Method No. 72: These dyes are products similar to the Acedronoles, Azoniles and other dyes for developing on acetate silk; in other words, they are bases, and most of them must be dissolved in hydrochloric acid for use in the dye bath. Azonine G is soluble in hot water without the use of hydrochloric acid. It is simply dissolved in boiling water, strained and added to the dye bath. The material is worked for about forty-five minutes at 60 to 70 deg. Cent. (140 to 160 deg. Fahr.), with the addition of 3 to 10 per cent of acetic acid. As the bath is not completely exhausted, it is best to use a standing bath. Azonines R, 2R, B and S are first dissolved in about one hundred times their weight of water and the particular quantity of hydrochloric acid given in Table VII, after which it is filtered through a cloth and added to the 60 deg. Cent. (140 deg. Fahr.) 30 or 40 to 1 dye bath.

TABLE VII

Hydrochloric Acid Required to Dissolve the Azonines

Weight of 34 deg. Tw. hydrochloric acid per pound of dyestuff:

Name	Ounces
Azonine R (1 pound)	16.0
Azonine 2R	13.0
Azonine B	9.5
Azonine S	9.5

In dyeing light shades with these dyes, or where there is trouble in leveling, an addition to the dye bath of 0.2 to 0.5 per cent hydrochloric acid, on the weight of the goods, may be advantageous. As the baths exhaust well, a standing bath is of no advantage. Azonines R, 2R and S may be mixed with each other in any proportion and give direct shades of yellow to orange without development. Azonine G also gives a direct yellow shade only and is not developed. Azonine B is colorless until developed.

Table VIII gives a list of the colors obtained with the various Azonines with the different developers:

TABLE VIII

Formula No. and Name — *Developer and Color*

Y1—4% Azonine G. Dev., Not developed. Lemon Yellow.
Y2—2% Azonine R. Dev., Phenol 2%. Golden Orange.
Y3—2% Azonine R. Dev., Resorcinol 2%. Red Brown.
Y4—2% Azonine R. Dev., B-naphthol 2%. Bright Scarlet.
Y5—2% Azonine R. Dev., Developed ON 2%. Dark Red.
Y6—2% Azonine 2R. Dev., not developed. Bright Yellow.
Y7—2% Azonine R. Dev., Resorcinol 2%. Red Brown (lighter than No. 3).
Y8—0.2% Azonine R. Dev., B-naphthol 1%. Scarlet (lighter than No. 4).
Y9—2% Azonine R. Dev., B-naphthol 2%. Scarlet (redder than No. 4).
Y10—2% Azonine R. Dev., Developer ON 2%. Deep Maroon.

*The author is indebted to B. K. Archer, of Kuttroff, Pickhardt & Co., for data on the Developed Azonines.

Formula No. and Name — *Developer and Color*

Y11—1.5% Azonine B. Dev., Developer 3%. Dark Blue.
Y12—1% Azonine S. Dev., Phenol 1%. Light Brown.
Y13—1% Azonine S. Dev., Resorcinol 1%. Very Dark Purple.
Y14—1% Azonine S. Dev., B-naphthol 1%. Darker and bluer. More purple than No. 13.
Y15—0.2% Azonine S. Dev., Developer ON 1%. Gray Blue.
Y16—3% Azonine S. Dev., Developer 3%. Black.

Method No. 72A: After dyeing, the colors, with the exception of Azonine G, may be diazotized according to the quantity of dyestuff used, with 2 to 4 per cent of sodium nitrite and 5 to 10 per cent of 34 deg. Tw. hydrochloric acid in a cold bath for fifteen to twenty minutes, and then rinsed. See also Methods No. 70A and 71A.

Method No. 72B: The color is then developed for twenty to thirty minutes in a 45 to 60 deg. Cent. (115 to 140 deg. Fahr.) bath, with from 1 to 3 per cent of developer and rinsed again. Azonine B requires about twice as much developer as the others. The developers are prepared by dissolving the phenol or resorcinol in hot water. *B*-naphthol is prepared by boiling together 100 parts of *B*-naphthol, 200 parts of Turkey Red oil and 2,000 to 3,000 parts of water.

Developer ON, which is possibly *B*-hydroxynaphthoic acid, is prepared by boiling together 100 parts of developer, 200 parts of sodium acetate and 2,000 to 3,000 parts of water. These solutions are then added directly to the developing bath. A light soaping after developing and rinsing will brighten the shade. The dyes do not stain cotton and are therefore useful for two-color effects on cotton acetate silk unions. See also Methods No. 70B and 71B.

Dyeing Acetate Silk with the Azoles

Method No. 73: The Azoles are another brand of dyes which come under the same classification as the preceding Azoniles, Acedronoles, etc. These products are now offered to give the following colors: Claret, red, scarlet, violet, prune, blue, tobacco, nigger-brown, green, olive and black. From these such mode shades as gray, sand, beige, drab, praise, mole and lilac may be produced. In applying these dyes the acetate silk should be scoured as in Method No. 5, when it is ready for dyeing.

Azoles AG, AR and AZ may be applied directly for useful shades; and while Azoles 6GL and R dye the acetate silk a greenish yellow, the direct shades are not fast and the dye must be diazotized and developed to give practical results. Azoles AG, AR and AZ, CB and GL are particularly fast to light. The developers recommended are phenol, resorcinol, *B*-naphthol and Azole Developer ON. The colors developed with phenol are too sensitive to alkalies to be of practical value, but when the phenol is combined with other developers useful shades are obtained. The shades developed with *B*-naphthol are not as fast to light as those obtained with resorcinol or Azole Developer ON. The Azoles have no affinity for cotton or vegetable fibers, and are therefore useful in unions where it is desired to leave the cotton white or dye it in contrasting shades with direct cotton dyes.

Azole 6GL is soluble in hot water, but the others require the quantity of 32 deg. Tw. hydrochloric acid shown in Table IX and 100 times their weight of boiling water. The Azoles may be mixed indiscriminately in the dye bath.

TABLE IX

Hydrochloric Acid Required to Dissolve the Azoles

Each 100 parts of Azole require:	Parts of 32° Tw. Hydrochloric Acid
AG	116
AR	116
AZ	174
B	87
T	87
D	87
R	87
CB	58

A slightly acid 30 or 50 to 1 dye bath is generally used. For light shades or to aid leveling, 0.2 to 0.5 per cent of 32 deg. Tw. hydrochloric acid, on the weight of the goods, may be added to the dye bath before seiving in the acid solution of the Azole as in Method No. 72. The goods are usually dyed at 55 to 65 deg. Cent. (130 to 150 deg. Fahr.) for about one hour, when the bath will be well exhausted. In applying heavy shades, 3 to 5 per cent on the weight of the goods, of sodium acetate crystals in solution may be added to the dye bath after dyeing for about half an hour to aid the exhaustion. They are then dyed for another half hour and rinsed.

Method No. 73A: The goods are developed in a 20 or 30 to 1 bath with 3 to 6 per cent sodium nitrite and 7 to 14 per cent of 32 deg. Tw. hydrochloric acid, as in Method No. 70A. They are then rinsed cold and are ready for developing.

Method No. 73B: Develop for one-half to three-quarters of an hour at 46 to 60 deg. Cent. (115 to 140 deg. Fahr) in a 20 or 30 to 1 bath containing 2 to 6 per cent of developer. Azoles B, T, D and CB require more developer in proportion than the other Azoles. The phenol and resorcinol are dissolved in hot water. The *B*-naphthol is dissolved by boiling it with 2 parts of Turkey Red oil and 20 to 30 parts of water. Azole Developer ON is dissolved in the same manner, only replacing the oil with sodium acetate crystals. The developers may be mixed in the same bath as desired. After developing, rinse, soap, rinse, acidulate with acetic or tartaric acid, hydroextract and dry. Pasic dyes may be used for topping if desired.

The U. S. Finishing Company has awarded contract to Albert Smiley, Pawtucket, for construction of a one-story, saw tooth roof, addition to the dyeing department of its Silver Spring Branch. The extension will be equipped with 47 dye jigs.

William A. Thompson, formerly with the Butterworth-Judson Corporation, has gone with the dye, chemical and color department of the Sherwin-Williams Company, in New York. He will handle chemicals, dyestuffs and intermediates.

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MIDSUMMER—A TIME FOR RETROSPECT AND FORWARD PLANNING

WHILE it is the general custom for corporations to arrange their affairs so that their fiscal year coincides with the calendar year it would, in a majority of instances, be more proper for both corporations and individuals to regard the annual cycle of business activity as beginning and ending with the period of midsummer dullness which prevails in nearly every industry.

At this time there is generally a let down in activity caused primarily by the extreme summer temperature and incidentally because of the prevalence of vacation periods among employers and employees. Such a period of comparative inactivity affords an excellent opportunity for a mental review of the events of past seasons and for the formulation of plans for the coming fall and winter.

Admittedly the past twelve months have been hard ones for the textile industry and consequently for the dyestuff industry which is so largely dependent upon textiles for its livelihood. The primal cause for lack of profits in both of these industries is unquestionably attributable in great measure to one common cause—great overproduction or potential overproduction and consequent competition of the keenest sort.

A review of general business conditions throughout the country during recent months shows unquestionably that industrial activity as a whole has been somewhat above normal. Bank clearings and railroad freight movements—the two most reliable indices of general commercial activity—show substantial improvement over corresponding figures for a year ago. The failure of the textile industry—along with a few others, such as shoes and leather, for instance—to share in this general improvement is somewhat of a phenomenon, but is, we believe, primarily accounted for by the factor previously mentioned, namely, great potential overproduction and consequent abnormal competition.

This potential overproduction, an inheritance from war-time expansion, will, of course, gradually right itself. Mills which find the pace too swift will be abandoned or converted to other purposes and this, together with the gradual increase in population, will eventually strike a balance in supply and demand. In the meantime, however, it behooves those who would conduct their businesses profitably to use something more than ordinary sales and manufacturing methods.

It is obvious that so long as dyestuff plants whose maximum productive capacity could supply perhaps twice the total domestic requirements continue to exist, there can be little expectation of a great enhancement in dyestuff prices. Assuming, therefore, that little increase in net revenue can be hoped for through an advance in price of the finished product, it is obvious that the concern which would increase its earnings must either lower its cost of production or secure for itself a greater proportion of the available business through the introduction of newer or better products or by means of more efficient sales methods.

Only a few of our dyestuff manufacturing concerns—and these, for the most part, are those with the largest resources in both cash and experimental equipment—have shown any great progress during recent months in the way of new and improved products. The average in this respect is, however, fairly well maintained, as at least two of these concerns have shown more within the last twelve months than at any time since the development of the American industry. Presumably these concerns will continue to progress in the way of new developments and will benefit accordingly.

The resources of the rank and file of smaller producers and dealers, however, do not permit of technical research on a large scale. It remains for them, therefore, to seek other methods of effort if they would maintain or extend their present volume of business. Unquestionably one of the most obvious of these methods, and one which is most likely to be successful, is intensive co-operation with and service to the consumer.

In many cases the salesman is little more than an order taker. In other cases where he may use intelligence and initiative himself he is not properly supported by the concern which he represents.

For example, the writer recently visited the main sales office of a fairly large dyestuff distributing organization and had the privilege of examining a large number of the salesmen's reports. Many of these were of the most stereotyped nature and showed little disposition on the part of the salesmen to inquire as to possible business opportunities or ways in which his house might possibly be of service to the customer. There were, however, some marked exceptions in which full details as to the class of work on which the customer was engaged were given and oftentimes samples of goods sent in together with a report as to

the nature of the dyestuffs which were being used thereon.

The writer asked the sales manager of this concern whether any efforts were being made to capitalize the information contained in these reports or any attempt made to secure more data from those salesmen who were apparently giving the matter little thought. Much to his surprise he learned that the reports were simply examined and filed and that the laboratory never made any active effort to assist the salesmen in securing business, but confined itself solely to the furnishing of samples and matches where such were specifically requested.

Unquestionably such an attitude is not designed to bring in any great amount of new business. If, on the other hand, wherever opportunity could be found, a consumer were to be furnished with samples and formulae showing wherein superior qualities could be obtained in the finished goods or where better money value could be had, it would, in many cases, lead to the opening of friendly relations which would eventually develop into active accounts bringing in much new business.

This is but one suggestion as to how new business may be obtained. Every executive and every salesman knows his own problems better than an outsider can possibly hope to know them. One thing is certain however, and that is that competition in the immediate future is going to grow no less keen, and that the concern which hopes to increase its volume of business or its margin of profit, must devise methods or products better than those of its competitors.

Advertising will help but, like all other methods of sales promotion, to be effective it must be intelligent and distinctive. The mere repetition of a firm's name and its products, while it unquestionably serves as a constant reminder, will not begin to produce the effect upon customers that carefully planned and regularly changed advertising copy will do.

The summer period gives all of us an opportunity to consider our plans for the coming fall and winter season. Those who give the matter intelligent and intensive thought and approach the fall season with a constructive plan completely outlined are almost certain to reap the benefit; while those who allow matters to follow the line of least resistance will in all probability suffer in proportion.

NEW PONTAMINE BLACK

E. I. du Pont de Nemours & Co. announce placing on the market Pontamine Fast Black LND which has been manufactured to meet the exacting requirements of the dyers of union goods.

This product will find its largest use in the dyeing of half-silk hosiery and union materials, and also on cotton and artificial silk, which is to be discharged to a pure white, or have discharge effects printed upon it.

Highs and Lows in a Busy Chemist's Life—Part VII

NO document of this sort is complete without some mention of the war, and probably a few personal experiences are typical of others.

First was the foreign companies' announcements of "business as usual" (until the dyes are gone), and they lived up to it very well without taking extreme advantage of their opportunities.

Then came offers of tinted salt, masquerading as dyes, at fabulous prices. Talk about the loaves and fishes! Here a few fragments were sold by the barrel.

At the same time came a hasty scurrying to dig out from the drug room and color shop all the dusty tins, dried-up kegs and caked pastes. First the ones possessing color, and then the odds and ends of chemicals. If they could not be used at the plant, good prices were received; and if half an inch of dust could be weighed in with them, so much the better. And how some bewailed their folly afterward, for parting with some of this "rubbish"!

It was not long before we had to begin to think up substitutes, and they were not all dyes either. Among the first colors to disappear were the silk white dyes for wool, and there was no substitute. Wood silk, however, was ready, and this vegetable fiber stayed white with many colors that stained real silk, and even with the wood dyes, logwood and fustic.

It was a lifesaver, and at the same time obtained a perfectly regular introduction to the textile industry. The name wood silk is really preferable to artificial silk, but it went out of use long before the days of glos and rayon.

The problems of war-time were new and varied. We tried every wool dye on our shelves, nearly, to see if it could possibly squeeze into a khaki formula, and then squeeze by the Government or Allied tests. So it went for almost every textile.

New products and new tests were also the order of the day. One, which taught a lesson, was a sample or two of gun-cotton, being checked for another laboratory. In testing for ether extract we used the same set-up as for extracting samples of worsted tops, etc., but obtained erratic results. It was finally found that cork stoppers, which had no appreciable effect when used over and over again in the apparatus on wool, did yield an appreciable resinous extract. It was not noticed when added to the 3 or 4 per cent extracted from wool, but became a very serious factor when added to the very small fraction of a per cent that is in gun-cotton. You know what eternal vigilance is, and it applies to laboratories as well as to nations.

Last but not least, in troubles or importance, were the makeshifts. For example, there was Aniline Black pigment. We had an improvised chemical plant in the middle of the laboratory, luckily near a flood drain. Inside the barrel was a cloth bag of the same size, and in the bottom of the barrel a plug.

The system of operation was to mix the proper ingredients in the proper manner in the barrel, and after the completion of the reaction to pull the plug. The bag obligingly retained all the pigment, while the liquor trickled out. By inserting the plug again, filling with water and stirring up, there was obtained a good wash, and after several repetitions of washing the batch was done. It was then lifted out in the bag and delivered to the color shop. All very extremely simple.

One step in the process was the gradual addition of a solution of chrome, the "gradual" being quite essential for success. For some reason, however, the assistant dumped in the whole solution at once; and, as we all know, uncontrolled oxidizing actions have a way of suddenly getting quite energetic. The whole black contents of the barrel arose in its wrath and flooded the laboratory. Luckily, as we have said, the "plant" was near a floor drain.

(Part VIII will appear in an early issue)

Practical Hints on the Production of Bright Colors on Textile Fabrics

Part XV

Continuous Dyeing Machine—Operation of the Plant—Advantages of the Plant—Dyeing Dark Shades Without Salt—Its Advantages

By RAFFAELE SANSONE

ALTHOUGH the continuous machine offers marked advantages for dyeing cotton fabrics with direct dyestuffs and for topping them with basic colors, it has to be employed with some caution to avoid serious results. On the continuous machine the dyeing and topping operations can be conducted in several ways. In one method work is started with a bath of regulated strength, maintained constant all through by additions of fresh dyestuff solution at regulated intervals; that is, before a too pronounced weakening can take place, resulting in a difference in the intensity of shade produced. The evenness of the shades and their regular brightening depends in this case on the diligence and capacity of the dyers, who have to be quite expert in making the additions of fresh dyestuffs and in regulating from the start the strength to be given to the different baths in the troughs of treatment, and to the stronger fresh dye solutions required for the additions. Certainly much depends in this case on several years of experience. The above has caused many dyers to prefer jig dyeing, as in this they know exactly what quantities of dyes have to be used for the treatment of each large piece of cotton cloth or of each two small pieces dyed together, and the number of turns to be given, with the resulting solutions for obtaining the desired ground color and topping.

With another method of dyeing on the continuous machine, the material is maintained from the start in contact with a large volume of dye bath of a fixed strength, caused to circulate through the plant at a speed regulated in accordance with the amount of dyestuff to be taken up by the cotton fabric. A similar arrangement is advantageous, as it avoids the danger of uneven dyeing in a same piece, or if the volume of the bath is sufficiently large, in a whole batch of material. The above arrangement requires, however, a very large supply of dyestuff bath when the goods are to be dyed in light shades. With medium shades more concentrated solutions can be used from the start in the case of the direct colors; this, however, only reduces to a certain extent the volume of the bath used.

When dyeing the direct colors on the continuous machine, however warm and concentrated may be the dye baths it is possible to produce shades beyond a certain intensity, and dark shades are still produced for this reason in many works on the jig. By making salt additions and bringing up the strength of the dye baths, the above inconvenience can be avoided, and shades of any required intensity produced much more conveniently than with ordinary jigs. The basic colors required for the topping operations, if not used with the necessary precautions, can result in a stronger covering of the cloth than may be desired, or may color this differently from yard to yard or at intervals. A way of obtaining even and convenient topping would be through adopting as dilute and long baths as possible, using some means for keeping these constant all through.

A plant that would offer special advantages for the dyeing of the grounds and their topping is constructed on the principle shown in Fig. 1.

Here is to be seen a long rectangular vat (H), placed on a lower floor of the mill. This vat is divided into four compartments, A, B, C, D, by special partitions, which between the first two compartments are perforated. Compartments A, B, and D are furnished with a closed steam heating coil, while compartment C is without and contains two upper taps for entering rinsing water and a lower tap for discharging this to the waste water main.

Vat H has four light squeezing arrangements supplied with lever and weight pressure, the upper roller

of which is of India rubber and the lower of copper-plated iron. At the beginning of the machine is a very effective entrance frame (E) for smoothing the cloth. At the end of the machine is a leading-out disposition, F, supplied with a rocking arm driven by a disc and iron rod, which folds the dyed and topped material onto a lower wooden stool, into a small wagon or onto a truck, as may be most convenient. The above squeeze rollers and plaiter F are driven by an electric motor.

Each compartment has a number of guide rollers, placed close to one another to assure thorough use of the baths of treatment and thorough penetration. Compartments A B, and D have a discharge tap leading in each case to a storage vat, or to the waste water main. Compartment B has a discharge tube (I) with a supplementary tap that causes a certain portion of the dye bath contained therein to fall into the lower cylinder K, from which it is carried to the lower portion of compartment A through centrifugal pump N and tube T. Here it then makes its way again into compartment B, forming a complete cycle.

Cylinder K is of fairly large size and can contain the bath necessary for the treatment of quite a large batch of material. In it is prepared a certain number of gallons of dye bath, regulated in accordance with the size of the batch to be treated, leaving room for the additions of fresh bath that may be necessary when reinforcing an old bath. All mixing operations are accomplished in this instance by a special agitator, turned from above by pinion wheels and the transmission, which also drives an agitator in the second cylinder (L) serving for the preparation, collection and circulation of the topping bath. Cylinder L receives the topping bath through tube J from the bottom of compartment D and delivers it again to the same compartment through the action of pump O and tube U. Transmission X is held in strong guards fitted to the ceiling of the cellar in which are placed the two cylinders K and L, and is driven from below by a leather belt and the electric motor M. The operatives descend to the cellar by an iron stairs (S).

The baths in cylinder K and L can be discharged either directly to the waste water main of the works, if this is sufficiently low, or through a special tube directly into the ground on the outside of the works, when this is not the case. Or they can be brought, through a special tap and tube on pumps N and O, after closing a tap on tubes T and U directly into collection cisterns, awaiting a later re-employment. The bottom of cylinders K and L contains a steam heating coil, to which steam is entered from without through a copper tap, and where all condense water formed is run out through a steam trap.

The small electric motors driving the centrifugal pumps N and O are supplied with a special speed regulator, through which the circulation of the bath in compartments A, B and D can be reduced or increased

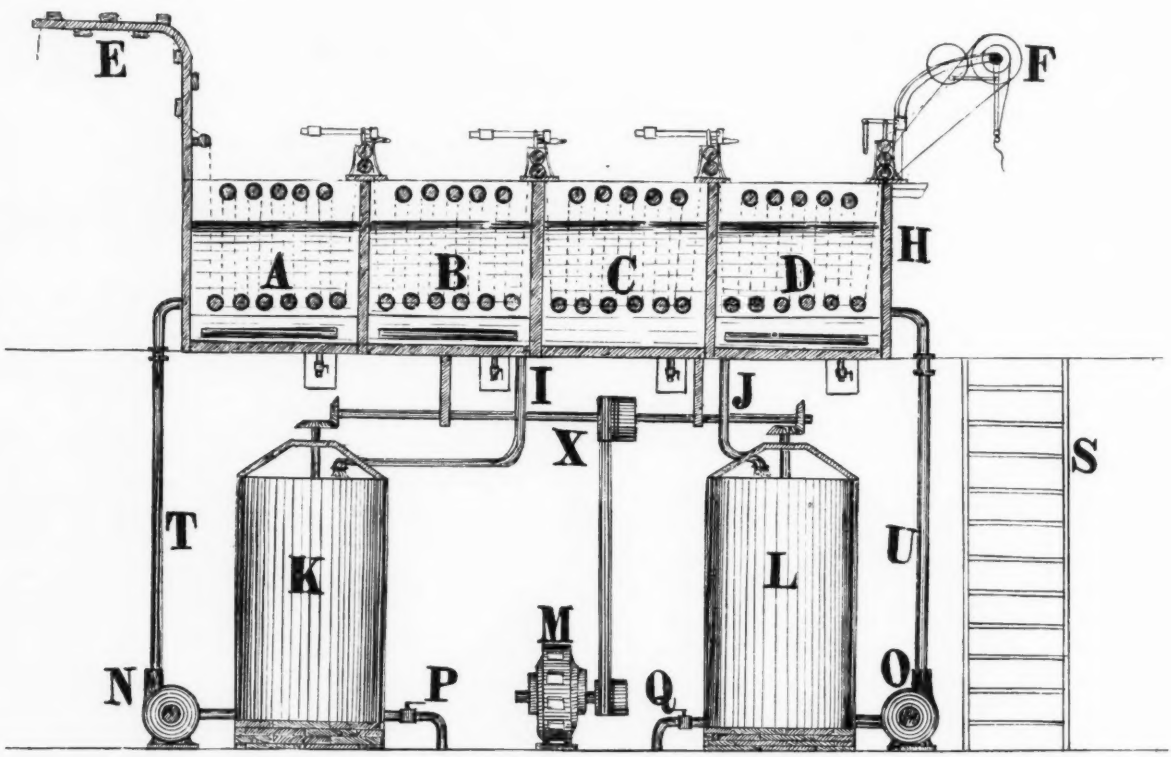

FIG. 1

within certain limits, in accordance with the nature of the material being treated, the strength of the dye bath used, the speed of the treatment of the goods, etc. The outflow of tubes I and J can also be regulated to keep the baths of treatment always at the same height.

Operation of the Plant

The operation of the above plant would require some experimenting before yielding the most satisfactory results. It can be conducted in different ways, one of which is as follows: The operators, after weighing and measuring the cloth to be dyed and topped, obtain instructions from the colorists regarding the shade, proportions and colors to be used, and when these have been obtained, consult special tables for calculating the number of gallons of bath to be prepared. These are based on the weight and yardage of cloth to be treated. They then introduce from upper taps, when fresh baths have to be prepared, the required quantity of water in cylinders K and L, starting the agitators after this, and turning on steam to bring the bath to the boil in the case of cylinder K, and to the most convenient temperature in the case of cylinder L.

When such operations have been conducted the operators drop in the coloring matter in each case, which is thus soon brought into solution; a piece of white filter paper can be spotted to observe if clear baths have been produced. They enter in the plant, meanwhile, a cotton tape, joining its end to the first piece to be dyed, and initiate the circulation of the dye baths in compartments A, B and in compartment D, reducing this to allow a thin stream of liquid to escape tubes I and J into cylinders K and L.

At this point a quantity of clean common salt is added in compartments A and B, and the plant is placed in motion to enter the cotton cloth. This passes down into compartment A, and an operator on each side of vat H smooths it as soon as possible of all plaits.

In compartments A and B the cotton cloth remains a certain amount of time, absorbing a fair quantity of the direct color. The slow circulation of the dye bath from cylinder K, the salt added to this, and the continual boiling facilitate the production of the deepest shades.

When the material leaves the second pair of squeeze rollers above compartment B it has thus been dyed in the required ground color, and the rinsing waters in compartment C prepare it for the topping that follows with a week bath in compartment D.

A special arrangement on the plant indicates through the ringing of a bell when a certain yardage of cloth has been treated, and when fresh dyestuff is to be added to cylinders K and L, which has been dissolved in small separate boilers heated by steam. The ringing of the bell is interrupted instantly by the moving of a short-hand lever or by other means, and only takes place again after the passage of another equal yardage of cloth. Should the bell ring when only a certain yardage of cloth has still to be treated, the operators regulate their additions to avoid preparing an excess of the strengthening baths.

Toward the end of the dyeing operations the operators fix to the end of the last piece entering, the cotton tape previously used, stopping the machine as soon as the beginning of this passes through plaiter F. In the topping bath the dyed cotton cloth can take up only a fixed quantity of the basic color, its permanence there being increased or reduced from the start by raising or lowering the upper guide rollers.

The above plant has several advantages, some of which can be indicated as follows:

1. By increasing the size of compartments A and B, and the number of guide rollers in these, it is possible to keep the material, although running through at a certain speed, a very fair period of time in contact with the boiling dye bath, so that the salt contained in this last has the time to fix the required quantity of the color.

2. Because of the very slow exit of the dye baths through tubes I and J very little of the salt contained in compartments A and B makes its way into cylinder K, so that the majority of this remains in the compartments all through the dyeing operations.

3. As it is possible to increase or reduce the speed of the passage of the cloth through the achine, different intensities of a same shade with one only dye bath may be produced.

4. The surface of the cloth in contact with the dye bath is strong, and allows the adoption of greater speed in the treatment.

5. The topping operations are conducted with a large volume of a weak solution of the basic color circulated through compartment D at a reduced speed, so that only a certain quantity of the dyestuff can be absorbed at a time.

6. To regulate the time in which the dyed pieces are in contact with the topping bath it is only necessary to lower or raise the upper series of guide rollers in compartment D.

7. Cylinders K and L can be employed to prepare fresh baths or to reinforce old baths.

(Part XVI will appear in an early issue)

CIBA INTRODUCES COMPLETE LINE OF DIAZOTIZING COLORS

Ciba Company, Inc., announces the readiness of a very complete line of diazotizing colors that are dischargeable with hydrosulphite. These products are brought out at a very opportune time, for there is a present and great demand for colors which have an excellent fastness to washing. In Diazo Fast Blues, Diazo Fast Browns and Rosanthrene fast colors dyers secure the important and sought-for features of fastness to washing, fastness to light and fastness to atmospheric influence.

DU PONT ANNOUNCES NEW ORANGE AND BROWN

Pontamine Fast Orange EG and Pontamine Fast Brown RK are two new colors recently announced by the Dyestuffs Department of E. I. du Pont de Nemours & Co.

Pontamine Fast Orange EG, a direct orange color of bright yellowish shade, is stated to be very fast to light and considerably faster to water, washing, perspiration, acids, and alkali than the average direct color. The announcement states, moreover, that it possesses fair fastness to laundry chlorine and that diazotization changes the shade very little.

When dyed on union material in a neutral bath the animal fibers are left practically unstained even at the boil. It is therefore used to a large extent for shading up the cotton in the one bath process for dyeing various types of union goods.

It is very soluble and level dyeing and may be recommended for use in any make of machine in ordinary use.

It may be dyed on either raw stock, yarns or pieces and it is extensively used with other direct dyestuffs of comparable fastness for the production of browns, tans, grays and mode and fancy shades of every description.

On silk, either pure or tin weighted, it is described as an extremely useful color, dyeing levelly and yielding shades of very good fastness to water and washing. It may also be used on artificial silk by the usual methods.

Pontamine Fast Brown RK is claimed to produce on cotton very reddish browns which are fast to light and very fast to chlorine for a direct color, and in addition shows much better fastness to washing, water, acids and alkali than most substantive dyes. It can be dyed with equally good results on raw stock, yarn or pieces, and on account of its good fastness to chlorine and washing, can be used on certain grades of material where good fastness is necessary.

Inasmuch as metals do not affect the shade and the solubility is very good, Pontamine Fast Brown RK can be recommended for any type of machine and is said to be especially useful for padding.

The animal fibers in unions, dyed in a neutral Glauber salt bath, are somewhat weaker and yellower than the cotton, while in speck dyeing the animal fibers are only slightly tinged.

This color is described as extremely fast to water, washing and light when used on either pure or tin weighted silk and on artificial silk dyed by the usual methods, the resulting shades are said to be very brilliant and very fast.

Pontamine Fast Orange EG and Pontamine Fast Yellow NN have somewhat similar properties so that combination of this new product with either or both of these colors gives a very wide range of oranges or browns, all said to possess excellent fastness.

Pontamine Fast Brown RK is not dischargeable.

CENTRAL BUREAU FOR SALE OF GERMAN DYESTUFFS IN BELGIUM

Under regime instituted by the treaty of Versailles, according to Assistant Trade Commissioner E. V. D. Wight, Belgium has received each year up until January, 1925, a certain quantity of dyestuffs which were divided among eight sales representatives, these representatives being the old pre-war connections of German dyestuff houses. Now that Germany bases reparation-in-kind deliveries upon articles of the Dawes plan, her dyestuff manufactures have decided to reorganize their sales procedure in Belgium. This is all the more logical in view of the fact that these German companies have associated themselves into a cartel for production of their colors.

As a result of the new sales policy, the eight Belgium sales representatives have united to form a "co-operative." All are taken in and work will be divided so that each partner will direct selling efforts to the trade with which he is most familiar: as, for instance, one will sell the woolen trade in Verviers, another the calf leather trade, another fur dyers, etc. Orders which they receive will be assembled in Brussels and communicated to the syndicate in Germany. In this way, with a common office and clerical help, overhead can be considerably reduced.

SEVENTH SOUTHERN TEXTILE EXPOSITION IN PREPARATION

The Seventh Southern Textile Exposition will be held in Greenville, S. C., in Textile Hall, November 1st to 6th, 1926. Notwithstanding the fact that the exposition is more than a year off, more space has been applied for than Textile Hall can accommodate.

These displays of textile machinery and supplies are among the most important industrial exhibitions held in America. Greenville is noted for this show which is held every two years.

For the 1926 event reservations have been made by a majority of the large machinery manufacturers of New England. Many applications have also been received from machinery makers and from textile supply and accessory concerns all over the eastern part of the United States.

ON X-RAY SPECTROGRAPHY

A twenty-page pamphlet, entitled "Applications of X-Ray Spectrography and Crystallography to Metallurgy and to Chemical Problems," has been issued by Adam Hilger, Ltd., London, England. The introduction treats of the varieties of X-rays and the kinds of photographic records obtainable with X-ray spectrographs. The remainder of the pamphlet deals with chemical and metallurgical applications in general, and includes an interesting bibliography giving abstracts of articles on X-ray crystal analysis that have appeared in American and foreign technical journals.

RECENT LITERATURE

Inks, Typewriter Ribbons and Carbon Paper. Department of Commerce, Bureau of Standards. Circular 95.

This booklet of thirty-two pages is a revised edition of a previous publication. It contains a discussion of the composition and methods of testing inks, typewriter ribbons and carbon paper. These methods, according to an abstract published with the contents, are not original, but have been compiled from a variety of sources, and modifications have been introduced when necessary. The introduction describes the early history and subsequent developments in the manufacture of inks. The chapters treat in turn of various types of writing inks and other inks, typewriter and other ribbons and carbon paper and include a complete and concise description of the methods of analyzing and testing the various inks whose properties are explained in the preceding sections. A bibliography of American and foreign literature on the subject is also included.

Elimination of Waste. Simplified Practice Recommendation of No. 27: Cotton Duck. Department of Commerce, Bureau of Standards.

Contains a report of the conference called at the request of a committee appointed by the Cotton Duck Association to study and submit recommendations in the matter of simplification of the weights and widths of sail and numbered cotton duck. A message from the Director of the Bureau of Standards and the Secretary of Commerce recommends the standard numbers and widths of sail and wide duck recognized by the industry. These numbers are tabulated. A list of the manufacturers, agents and distributors and consumers and associations that have accepted them is included in this booklet.

Reclamation of Gasoline Used in Dry Cleaning. By C. C. Hubbard, Research Fellow, National Association of Dyers and Cleaners. Department of Commerce, Bureau of Standards. Technologic Papers of the Bureau of Standards, No. 280. Illustrated.

This paper outlines and discusses briefly the processes that have been used in the dry-cleaning industry for the "purification" and recovery of gasoline from the material that has become dirty in use. Results are reported of experimental studies in the laboratory and in dry cleaning plants and of large scale tests in dry cleaning plants, using activated carbon and an aqueous solution of trisodium phosphate. A process is recommended for the rapid and economical purification and recovery of gasoline in large or small plants.

Flammable Liquids. National Fire Protection Association.

Contains a section dealing with dry cleaning and dry dyeing plants, outlining regulations for safeguarding such plants. These regulations include general requirements for fire protection and describe types of vacuum systems approved, recommending location of plants, construction of buildings, ventilation, lighting, heating and power; describe proper installation of tanks, purifiers, clarifiers, pumps, pipe manifold for handling solvents, washers, drying tumblers, extractors, separators and stills; describe approved method of scouring and equipment and general operating requirements, fire protection equipment and air conditioning.

Report of Board of Visitors to Bureau of Standards of the Department of Commerce. Miscellaneous Publication of the Bureau of Standards No. 63.

The value of basic research, aimed at the acquisition of knowledge without immediate utilitarian objectives, is emphasized in the report of the Visiting Committee of the Bureau of Standards. The committee expresses the belief that such work should be undertaken by the Bureau to a greater extent than has been done in recent years.

The report also calls attention to the value of industrial research—a value more generally realized. Numerous examples of savings effected by such research at the Bureau of Standards are given.

Both types of research, the committee believes, are essential to the welfare and industrial prosperity of the nation. The economies resulting from them are often very considerable, and are necessary if America is to maintain its present high wage rates and high standards of living in the era of intense industrial competition upon which the world now appears to be entering.

The desirability of having research conducted by a national institution is emphasized. Its value depends on the extent to which the results are utilized rather than upon the cost of the work, and a national laboratory such as the Bureau of Standards can afford to give its results freely to all who can profit by them, whereas a private laboratory cannot always be expected to do so.

This report contains much information about the nature and work of the Bureau of Standards that is not generally known, even in the industries which the Bureau serves.

The Aberfoyle Manufacturing Company, Chester, Pa., is reported planning the establishment of a branch mercerizing plant in the South, probably in North Carolina.

Dyestuff Tables

A List of the Principal American-Made Colors with Their Properties and Fastness Characteristics

[NOTE: The publishers of The REPORTER have secured more or less complete data in regard to many of the dyestuffs which are at the present time being manufactured in the United States. As the following material has been brought together from various sources, they realize that errors are quite likely to appear, and it is earnestly requested that any readers who find errors or who are able to give additional information will communicate with the publishers in order that this material may be made as correct, complete and up to date as possible. These tables will appear serially until all colors upon which we have data shall have been covered. The publication of this data was begun in the issue of March 10, 1924.]

FORMYL VIOLET S4B

(Schultz No. 530)

COMPOSITION: Triphenylmethane.

SPECIALLY SUITABLE FOR: Wool and union dyeing.

USUAL METHOD OF DYEING: On wool, with Glauber salt and sulphuric acid; on unions, with Glauber salt.

SHADE: Between S-527 and S-528.

SHADE BY GASLIGHT: Much redder.

SOLUBILITY: Liable to bronze.

LEVEL: Inferior to S-528; suitable for use as a shading color on navies.

EXHAUSTS: Well.

FASTNESS TO:

Acid: Good.
Alkali: Good; better than the average. Excels S-527.
Carbonizing: Fast.
Fulling: Best of the acid violets; inferior to the alkali violets.
Ironing: Somewhat duller; does not stain white.
Light: Moderate, though better than S-528.
Perspiration: Fast.
Rubbing: Crocks somewhat.
Steaming: Fast.
Sulphur: Fast.
Washing: Fair.

DYED BY OTHER METHODS: The color is only slightly affected by chrome and is used for brightening navies dyed with S-181 top chrome. Dyes chromate O. K.

ON UNIONS: Gloria: Both fibers dyed equally in an acid bath. Dyes wool in a neutral bath; the best of the acid violets. On cotton-wool unions the cotton is tinted violet in a neutral bath; in an acid bath the cotton is tinted slightly bluish.

ON OTHER MATERIALS: On silk: Dyed with boiled-off liquor and sulphuric acid; fairly fast to water on this fiber. On paper, lakes lithographic inks: Suitable.

DISCHARGING: Dyed on wool, the shade cannot be discharged with tin crystals, but discharges white with zinc dust.

COMPETING PRODUCTS: Made in the United States by Cincinnati Chemical Works, under name of Acid Violet; Chemical Company of America, under name of Acid Violet 3B; Newport Chemical Works, Inc., under names of Acid Violet 5B and Acid Violet S4B; Garfield Aniline Works, under name of Acid Violet S4BM; E. I .du Pont de Nemours & Co., under name of Pontacyl Violet C4B; National Aniline & Chemical Company, under name of Wool Violet 4BN.

FAST ACID VIOLET 10B

(Schultz No. 528)

COMPOSITION: Triphenylmethane.

SPECIALLY SUITABLE FOR: Wool.

USUAL METHOD OF DYEING: Glauber salt and sulphuric acid.

SHADE: Very bluish violet.

SHADE BY GASLIGHT: Redder and duller.

SOLUBILITY: Good.

LEVEL: Dyes level.

FASTNESS TO:

Acid: Fast.
Alkali: Fast.
Carbonizing: Fast.
Light: Moderate; same as average acid violet. Suitable for ladies' dress goods.
Perspiration: Fast.
Steaming: Stands dry steaming.
Sulphur: Fast.
Washing: Fairly fast for acid dye.

OTHER PROPERTIES: Used in combination shades on yarn and pieces.

DYED BY OTHER METHODS: The shade is not affected by chrome to any extent, the after-chromed shade being somewhat faster to milling.

ON UNIONS: Silk-effect threads are stained. Cotton-effect threads are left white in an acid bath.

ON OTHER MATERIALS: Silk: Dyed in bath of boiled-off liquor broken with sulphuric acid. Not fast to water on this fiber.

PRINTING: Suitable.

COMPETING PRODUCTS: Made in the United States by Newport Chemical Works, under name of Acid Violet 10B; Chemical Company of America, under name of Fast Acid Blue XL; E. I. du Pont de Nemours & Co., under name of Fast Violet 10B.

ACID FAST VIOLET BG

(Schultz No. 531)

COMPOSITION: Triphenylmethane.

SPECIALLY SUITABLE FOR: Wool and silk.

USUAL METHOD OF DYEING: Glauber salt and sulphuric acid.

SHADE: Similar to S-528.

SHADE BY GASLIGHT: Slightly redder.

SOLUBILITY: Very soluble.

LEVEL: Dyes level.

EXHAUSTS: Well.

PENETRATION: Very good.

FASTNESS TO:
Acid: Fast to acetic; greener with sulphuric.
Alkali: Fast.
Carbonizing: Shade returns on neutralizing.
Fulling: Whites are stained; shade goes lighter.
Light: Moderate; equals S-528, inferior to S-530.
Rubbing: Fast.
Steaming: Fast to moderate steaming.
Washing: Bleeds off.

ON UNIONS: Gloria: Both fibers dyed a uniform shade in an acid bath (no Glauber salt).

COMPETING PRODUCTS: Made in the United States by National Aniline & Chemical Company, under names of Acid Fast Violet BG and 12B.

ALKALIE BLUE
(Schultz No. 536)

COMPOSITION: Triphenylmethane.

SPECIALLY SUITABLE FOR: Wool.

USUAL METHOD OF DYEING: In alkaline bath with borax. Develop with sulphuric acid in a fresh cold bath.

SHADE: Bright pure blue.

SHADE BY GASLIGHT: Slightly redder.

FASTNESS TO:
Acid: Fast.
Carbonizing: Will not stand.
Fulling: Moderate.
Light: Moderate.
Rubbing: Crocks somewhat.
Steaming: Will not stand.
Sulphur: Will not stand stoving.
Washing: Moderate.

DYED BY OTHER METHODS: Dyes wool in a neutral bath.

ON UNIONS: Does not leave silk or cotton white.

ON OTHER MATERIALS: Silk: Dyed in a soap bath and developed in a fresh bath of sulphuric acid cold. Fast to water and acids on this fiber. Moderately fast to light.

PRINTING: Can be printed on cotton with tannin.

COMPETING PRODUCTS: Made in the United States by Calco Chemical Company under name of Alkali Blue; Sherwin-Williams Company, under name of Alkali Blue; National Aniline & Chemical Company, under names of Alkali Blue B and R; N. Y. Color & Chemical Company, under names of Alkali Blue 6B and 3R; Albert David Chemical Company, under names of Alkali Blue Conc., Alkali Blue Pulp Mono-Sulphonic, and Alkali Blue R.

SOLUBLE BLUE
(Schultz No. 539)

COMPOSITION: Triphenylmethane.

SPECIALLY SUITABLE FOR: Wool.

USUAL METHOD OF DYEING: Glauber salt and sulphuric acid.

FASTNESS TO:
Acid: Fast.
Carbonizing: Fast.
Ironing: Fast.
Light: Moderate.
Rubbing: Crocks badly.
Steaming: Stands moderate steaming.

DYED BY OTHER METHODS: Used to brighten logwood.

ON OTHER MATERIALS: Silk: Dyed with boiled-off liquor and sulphuric acid. Cotton: Can be dyed with stannate of soda.

DISCHARGING: Zinc gives a temporary white.

COMPETING PRODUCTS: Made in the United States by National Aniline & Chemical Company, under names of Pure Soluble Blue A and B; Albert David Chemical Company, under names of Soluble Blue 2B, 2G, 2R and 5R; Calco Chemical Company, under names of Soluble Blue 3G and 5R; N. Y. Color & Chemical Company, under names of Soluble Blue BB, R, 3R and Water Blue 6B.

ALPHAZURINE 2G
(Schultz No. 543)

COMPOSITION: Triphenylmethane.

SPECIALLY SUITABLE FOR: Wool and silk.

USUAL METHOD OF DYEING: Glauber salt and sulphuric acid.

SHADE: Greenish blue.

SHADE BY GASLIGHT: Brighter and clearer.

SOLUBILITY: Good.

LEVEL: Excellent.

EXHAUSTS: Very slowly; S-545 better.

FASTNESS TO:
Acid: Fast to acetic (1-10). Sulphuric (1-10) turns shade yellower. On neutralizing shade returns.
Alkali: Greener with ammonia. S-543 better than S-545.
Carbonizing: Fast with chloride of alumina.
Fulling: Poor; changes shade and bleeds into whites; after-chromed slightly better.
Ironing: Fast.
Light: Five days, no change in shade; fourteen days, shade changes.

(Continued on page 478)

Technical Notes from Foreign Sources

Separation of Wool Fats from Wash Liquors

OILY or fatty substances are separated from, for example, wool scouring liquors by injecting a large quantity of air (approximately 200 volumes of air per unit volume of liquor) in the form of minute bubbles and maintaining a sufficient depth of froth upon the surface to insure the adhering liquor draining away. The depth of liquor through which the bubbles rise should be such that sufficient time is permitted for them to entrain the particles of fatty matter, with the formation of a satisfactory froth, and should be increased in the case of emulsions of low fat content.

The surface of the liquor should be relatively quiescent to prevent remulsification, and the temperature should be maintained at or but slightly above the melting point of the fat. The temperature of the froth is controlled by fine sprays of cold water, which also serve to wash out the adhering liquor and stabilize the froth.

For example, wool scouring liquor is cooled to a temperature varying between 32 and 38 deg. Cent., and is then caused to flow through a series of aeration boxes, the depths of liquor and froth being about 1 foot and 2 feet, respectively. The froth is continuously sprayed with cold water and is periodically removed and broken down, the wool fat being left, after removing the water and impurities in the form of a thick cream. The aerated liquor, after passing through a settling device to effect the removal of the sludge, may be employed for the treatment of further quantities of wool, the scouring process thus becoming a continuous one.

[For further details see British Patent No. 205,833.]

Oxycellulose in Cotton

Investigation of the oxidation of bleached and scoured cottons under controlled conditions by means of chlorine water, acid solutions of potassium permanganate, and acid and alkaline hypobromite and hypochlorite solutions shows that the resulting oxycellulose falls between two extreme types, one characterized by a high affinity for Methylene Blue and the other by strong reducing properties as measured by the copper number. The production of either type in cotton materials results in decreased tensile strength, but fabric which contains the former type of oxycellulose suffers much less loss of weight during scouring than does material which contains the latter type.

In technical overbleaching by means of hypochlorites, the type of oxycellulose produced is largely dependent on the acidity or alkalinity of the bleaching liquor. Alkaline bleach liquors favor the formation of oxycellulose which has a high Methylene Blue number and a low copper number, but acid bleach liquors give rise to oxycellulose which has a low Methylene Blue absorption and high reducing power.

Chlorine water attacks cotton but slowly in the dark, whereas under similar conditions an alkaline solution of hypobromite oxidizes cotton rapidly. Hypobromites are well adapted for the preparation of standard oxycellulose products. Oxycelluloses which are produced by means of alkaline hypobromite, acid permanganate and chlorine water have low, moderate and high copper numbers, and high, moderate and low affinities for Methylene Blue, respectively.

The yellowing of oxidized cotton on heating or treatment with alkali is closely associated with an enhanced copper number; a low copper number indicates permanence of the white of a bleached fabric. The affinity of bleached cotton for direct cotton dyestuffs is associated with the Methylene Blue absorption; bleached fabric which has an uneven absorption for Methylene Blue has an uneven affinity for direct cotton coloring matters, such as Diamine Sky Blue FF, etc.

[For further details see Jour. Tex. Inst., 1925, 13-52T.]

Mildew on Linen Goods

The cause for the appearance of mildew on linen goods is to be found in certain micro-organisms that attack the fiber. These bacteria, together with the action of certain enzymes, destroy the fiber. According to the degree of growth of these micro-organisms, spots are formed on the fiber which are thereafter colored gray, light green, red or brown. As is known, mildew will form easily when washed goods are allowed to remain in the moist condition in places which are not properly ventilated.

So far as cotton goods are concerned, the processes which takes place in the formation of mildew are rather well understood, but this is not the case with linen goods and the bax fiber. Investigations are now under way which have for their purpose the exact determination of the mechanism of the formation of mildew on linen ware.

[Deutsche Leinen Ind., 1924, page 633.]

Analytical Determination of the Amount of Color in Dye Liquors

The proportion of coloring matter that is contained in a dye liquor or the degree of exhaustion of the dye bath is determined in practice by making a test dyeing and comparing the intensity of the color thus obtained with standards. Methods that are based on physical

processes appear to be less accurate than those that depend on chemical operations.

Among the methods used for this purpose that are of chemical origin may be mentioned those that depend on the reduction of the coloring matter with the aid of chloride of titanium or sodium hydrosulphite, which are mostly employed. An investigation was made of these methods and the comparative results are given in the original article on this subject contained in Helvetica Chim. Acta, Vol. 7, pages 507 to 513. It was found that the titanium chloride method is to be preferred.

If it is found that the coloring matter is hard to reduce, then an excess of the titanium salt is employed, then an excess of Methylene Blue is added, and the excess of the latter dyestuff is determined by means of a back titration with chloride of titanium. The method gives the same satisfactory results when Benzopurpurine 4B is used as the coloring matter.

When chrysophenine is employed, while the results are better than those which are obtained by the hydrosulphite method, the accuracy thereof cannot be any more than merely comparative. The tests were generally carried out in a tartaric acid medium in the case of benzoazurine and pure diamine blue. They may also be carried out in a sulphuric acid solution. When hydrosulphite is employed, a solution of this salt, which has been stabilized with acetone and ammonia, is employed.

Vat Dyeing of Piece Goods

The dyeing of piece goods with the aid of vat coloring matters may be divided into three operating stages. In the first place, there is the preliminary treatment of the goods, then the coloring proper, and, thirdly, the after-treatment. The preliminary treatment of the goods consists generally in the bleaching operation or at least in the thorough wetting down of the fabrics. The pieces, which are advantageously 500 meters long, are fastened to leaders about 3 to 4 meters long, so that the ends are well dyed.

There are two processes which are generally employed in dyeing the goods. In the first process the fabrics are dyed at a temperature of 30 to 60 deg. Cent., and then there is what is known as the primary vat process.

Fifty kilograms of the fabric are dyed with a 10 per cent Indanthrene Golden Orange G double-strength paste. The coloring matter is mixed with a textile oil, and then boiling-hot water is poured over the paste. The color bath of 200 to 300 liters capacity is mixed with approximately 2 liters of a 40 deg. Be. solution of caustic soda and 500 grams of hydrosulphite. The coloring matter is sifted through a fine sieve and added to the dye liquor, and then a further quantity of hydrosulphite (500 grams) is added and the dyestuff is allowed to be reduced preparatory to bleaching. A pure carmine red color is obtained.

At this point the actual dyeing operation commences. Before passing the piece goods through the color bath for the second time, the remainder of the coloring matter and a further 500 grams of hydrosulphite are added to the dye bath. The goods are finished after from six to ten passages through the dye liquor. The temperature at the start is 30 deg. Cent. and it is gradually raised as the dyeing proceeds, finally reaching the boiling point of the dye liquor. Care must be taken to see that no oxidation takes place during the dyeing operation, and that no spots are formed on the goods. This indicates a lack of sufficient hydrosulphite in the dye bath.

In another process 50 kilograms of piece goods are dyed with 5 per cent of Thioindigo Blue 2G Powder. One kilogram of the dyestuff is made into a paste with a suitable textile oil, and then 5 liters of boiling-hot water are poured over the same. Thereafter 150 c.c. of caustic soda lye of 40 deg. Be. concentration are added and 120 grams of hydrosulphite, and the volume is made up to 1 liter. The temperature at which the color is developed is 45 deg. Cent. and the color of the liquor is a yellowish brown. The dye liquor, amounting from 200 to 300 liters, is mixed with 1 liter of a sodium hydroxide solution of 40 deg. Be. concentration and the process is carried out as described above. An additional 500 grams of hydrosulphite are added during the progress of the dyeing operation. The temperature at which dyeing takes place is only 20 to 30 deg. Cent.

The after-treatment of the dyed fabric is somewhat as follows: The first step in the process is to wash the fabrics very thoroughly with water until complete oxidation takes place, which means that the proper shade of color is allowed to develop. Oxidizing agents are advisably employed in order to increase the speed of the process. When yellow tones are being developed, it is customary to employ potassium dichromate. Sodium perborate may be used in all other cases. The oxidizing agents are added in solution to the third or fourth wash bath. The final treatment is in a boiling-hot liquor containing soap and soda. This treatment is essential with all sorts of vat dyestuffs, and it is only after such treatment that the fastness and brilliancy of the vat shades are first developed.

When oxidizing darker shades, such as Marine Blue and Blacks, certain definite stages must be gone through. The goods are permitted to remain in contact with the air for a definite period of time. Then they are very thoroughly washed. In order to obtain a clear, solid color tone it is advisable to add from 1 to 2 grams of hydrosulphite to the last wash liquor. In order to obtain a black, the dark gray color that is first obtained must be subjected to after-treatment with chloride of lime, chlorine or diazotization and development in order to be converted into a black.

[Deutsche Faerber Zeitung, 1925, 57-8.]

DYESTUFF TABLES

(*Continued from page 475*)

Perspiration: Fairly fast.
Rubbing: Fast.
Scouring: Poor if soda is used.
Steaming: Stands a dry steaming.
Sulphur: Fast.
Washing: Moderate; scours off.
Water: Moderate.

OTHER PROPERTIES: Used in piece dyes on ladies' dress goods.

DYED BY OTHER METHODS: Goes well onto the wool in a neutral Glauber salt bath. It is, however, inferior to S-545 in this respect. Suitable for shading top chrome colors. The after-chromed dyeings show a better fastness against white wool than the direct dyeings.

ON UNIONS: Cotton-effect threads remain white. Cotton-silk unions: Dyed in acid bath, the cotton is hardly tinted.

ON OTHER MATERIALS: Silk: On silk beautiful shades are produced which are not very fast to water. In this respect the S-545 is considerably better. Gloria: Both fibers dyed evenly. Paper: Suitable in pulp or for dipping or brushing.

PRINTING: Prints on wool or gloria, but the fastness of the printed shade to water is very moderate.

DISCHARGING: Discharges are produced with hydrosulphite. Not dischargeable with tin crystals and may be used for color discharges.

COMPETING PRODUCTS: Made in the United States by National Aniline & Chemical Company, under name of Alphazurine 2G.

PATENT BLUE A

(Schultz No. 545)

COMPOSITION: Triphenylmethane.

SPECIALLY SUITABLE FOR: Wool and silk.

USUAL METHOD OF DYEING: Glauber salt and sulphuric acid.

SHADE: Purer blue than S-543.

SHADE BY GASLIGHT: Slightly greener.

SOLUBILITY: Excellent.

LEVEL: Excellent.

EXHAUSTS: More quickly than S-543.

PENETRATION: Excellent.

FASTNESS TO:
Acid: Moderate to mineral acids.
Alkali: Inferior to S-543.
Carbonizing: Fast.
Cross-Dyeing: Good when resisted.
Fulling: Excels S-543; better after-chromed than direct.
Light: Same as S-543; used in ladies' dress goods.
Potting: Moderate.
Rubbing: Fast.
Scouring: Excels S-543; poor if soda is used.
Steaming: Moderate fastness.
Street Dirt: Moderate.
Sulphur: Fast.
Washing: Excels S-543.

SENSITIVE TO METALS, LIME: Shade goes duller with copper and iron. Shade is duller with chrome.

DYED BY OTHER METHODS: Dyes wool in neutral bath a fuller shade than S-543.

ON UNIONS: S-545 has more affinity for cotton than S-543. Gloria: Both fibers the same shade (fast to perspiration).

ON OTHER MATERIALS: Paper: Suitable for the calender or for coating or dipping. Lakes: Suitable. Silk: Dye in bath of boiled-off liquor and sulphuric acid below the boil; after-treated with tannic acid, faster to water. Feathers: Suitable.

PRINTING: O. K. for printing, though printed shade is not fast to water.

DISCHARGING: White with hydrosulphite.

COMPETING PRODUCTS: Made in the United States by National Aniline & Chemical Company, under name of Alphazurine A; Chemical Company of America, under names of Brilliant Blue A and B; Newport Chemical Works, Inc., under name of Patent Blue A; E. I. du Pont de Nemours & Co., under names of Pontacyl Brilliant Blue A and Pontacyl Brilliant Blue A for Lakes.

VICTORIA BLUE R

(Schultz No. 558)

COMPOSITION: Triphenylmethane.

SPECIALLY SUITABLE FOR: Cotton.

USUAL METHOD OF DYEING: On a tannin mordant.

FASTNESS TO:
Cross-Dyeing: Fair topped with tannin.
Washing: Good topped with tannin.

ON OTHER MATERIALS: On wool: Dyed with Glauber salt and sulphuric acid; fairly fast to sulphur; fast to street dirt and ironing. On silk: Dyed with boiled-off liquor and acetic.

DISCHARGING: Chlorate and soda suitable. Zinc discharge: Color returns partly, on exposure to air.

COMPETING PRODUCTS: Made in the United States by E. I. du Pont de Nemours & Co., under name of Victoria Blue R.

A special protocol prohibiting the use of poison gas or bacteria in warfare, sponsored by the American delegation, was unanimously adopted on June 10th by the International Conference for Control of Traffic in Arms, at which representatives of forty-four nations are convened in Geneva. Ratifications of the protocol, to which all nations will be invited to subscribe, and which will remain open for signature until September 30, 1926, are to be sent to the French Government. The protocol will be operative for each signatory from the date of ratification, as regards other powers which have ratified.

CIBA INTRODUCES NEW SWISS DYESTUFF

The Society of Chemical Industry in Basle has, under the name of Alizarine Sapphire Blue R, placed a new acid dyeing wool dyestuff on the market which is distinguished for its fastness to light and to perspiration. The dye belongs to the class of the Acid Alizarine colors and is dyed with Glauber salt and sulphuric acid. The new product is specially recommended for shades fast to light on piece goods, carpet yarns, etc. It is further suitable for ordinary and weighted silk. It is fast to decatizing, carbonizing and stoving; cotton remains undyed.

NATIONAL BRILLIANT WOOL BLUE B

A leaflet recently issued by the National Aniline & Chemical Company describes the properties of National Brilliant Wool Blue B. This color, it is claimed, yields very bright shades of blue possessing good fastness to washing, fulling and decatizing, and excellent fastness to stoving and perspiration. Its resistance to the action of metals, together with excellent solubility, makes it well adapted for use in all types of machines.

This color is said to exhaust well from a neutral bath and therefore is well suited for the shading of wool and silk in the dyeing of union materials.

Its fastness to washing and milling on aftertreatment with chrome permits its use for the shading of chrome colors.

National Brilliant Wool Blue B is also said to be well suited for the dyeing of glorias, vegetable and chrome tanned leather, straw braid, feathers, vegetable ivory, paper, jute and hemp, and for the printing of wool and silk piece goods.

PONTAMINE FAST BROWN RK

The dyestuffs department of E. I. du Pont de Nemours & Co., Inc., has just placed on the market Pontamine Fast Brown RK, a direct color which produces on cotton a bright reddish brown which is very fast to light. It also possesses very good fastness to chlorine and considerably better fastness to water, washing, acid and alkalies than the average substantive color. It is very soluble and level dyeing, and may be used on cotton in any stage of manufacture.

Because of its fastness properties Pontamine Fast Brown RK is recommended for all kinds of material which must show greater fastness than can be obtained with the usual direct colors. It is especially useful on account of its good fastness to chlorine and washing for shirtings, dress goods, etc., on which these properties are desired but where, for any reason, vat colors cannot be considered. In combination with Pontamine Fast Orange EG, Pontamine Fast Yellow NN, Pontamine Yellow SX, SXR and SXG, all of which possess somewhat similar properties, a very good range of oranges, browns, etc., may be produced.

Pontamine Fast Brown RK may be dyed on silk both pure and tin-weighted, the resulting shades being extremely fast to water, light, washing and perspiration. On artificial silk dyed by the usual methods the shades are very brilliant.

On union materials Pontamine Fast Brown RK dyes wool and silk weaker and yellower than the cotton, but when used as a speck dye the animal fibers are stained only slightly.

KLAUDER-WELDON INTRODUCES NEW MACHINE FOR HOSIERY DYEING

After many months of research, investigation and actual experiment, the Klauder-Weldon Dyeing Machine Company, of Bethayres, Pa., has secured patents on a machine designed to greatly simplify and improve hosiery dyeing. It is said that the new machine will be sold at lower prices than any hosiery dyeing machine built by this concern.

While the new machine is built for all grades and conditions of hosiery dyeing, it is especially suited for dyeing silk or light-weight sheer hosiery now in vogue.

Tre machine is built of Monel-metal, well calculated to withstand the wear and stress of any amount of work. A special feature is the cylinder constructed of Monel-metal, so built as to avoid the exposure of rough edges of sheets, cast metal surfaces or bolt or rivet heads. The perforating is so planned that the goods will not come in contact with perforated section at any time in the revolution of the cylinder, nor be damaged by reason of such contact.

The use of nets is entirely dispensed with, and the control of the mechanical action of the operation on the fabric is placed entirely in the hands of the dyer. He can, by raising or lowering the liquor level, assure himself of quality work by increasing or decreasing the mechanical action necessary.

Because of these great improvements and lowered costs, the Klauder-Weldon Dyeing Machine Company is preparing to increase its production to care for greatly increased demands.

COMMITTEE CONSIDERS SEVENTY-FIVE CHEMICAL COURT ENTRIES

Between seventy-five and eighty applications for the Court of Chemical Achievement to be held in conjunction with the Tenth Exposition of Chemical Industries at the Grand Central Palace, New York, during the week of September 28th to October 3rd have been received, and most of them have been submitted to the Approval Committee of the American Chemical Society. Preliminary consideration of some of the applications, which are for new chemical products, instruments, equipment, and new processes developed in the United States during the past few years, indicate that a few will probably not be accepted. The majority, however, are expected to secure admission, although the final decision one way or the other rests with the American Chemical Society Committee.

Reports from the Chemical Exposition management

state that the entire first and second floors of the Grand Central Palace have been contracted for by exhibitors, while a goodly portion of the third floor is already taken. Of course, the Court of Chemical Achievement, which will be on the third floor, will take a large part of this space and will attract many visitors to this floor. Total exhibitors to date indicate that this year's Chemical Exposition will be one of the largest industrial expositions ever held in the United States.

NORTH CAROLINA TEXTILE SCHOOL GRADUATION

At the graduating exercises of the Textile School of North Carolina State College, diplomas and degrees were awarded to twenty-seven textile students who had completed their courses.

The Student's Medal of the National Association of Cotton Manufacturers was awarded to J. E. Weber, of Morganton, N. C. This medal was presented by J. M. Gamewell, vice-president of the North Carolina Cotton Manufacturers' Association and general manager of the Erlanger Mills, Lexington, N. C.

During the commencement exercises, a $250,000 gymnasium was dedicated to the memory of Lieutenant Frank Martin Thompson who was killed in action in France during the World War. Lieutenant Thompson was a textile graduate with the class of 1910.

The majority of the graduates are from North Carolina, but other States represented are South Carolina, Georgia, Tennessee and Texas. The foreign countries represented are China, Japan and India.

DECLINE OF FRENCH CHEMICAL STOCKS

The stocks of the leading chemical producing companies in France, Trade Commissioner D. J. Reagan, Paris, advises, have continued the decline began the latter part of 1924, although in many instances the results of their operations for the year 1924 have been favorable, and in most cases the former dividends have been maintained, with the exception of a few companies whose capitals have been heavily increased.

This depreciation, which, in some instances, has amounted to almost 50 per cent from the high of 1924, reflects the general sagging tendency of the Bourse, owing to the lack of confidence in industrial circles and to the fear of competition, particularly in the dyestuffs group, although at present French dyestuffs manufacturers are reported to be supplying 95 per cent of the colors consumed in France, whereas in 1913 this proportion of French consumption was imported.

NEW HOME FOR JOHN D. LEWIS

The business of John D. Lewis, manufacturer and importer of dyestuffs and chemicals, moved on July 1st to its new office and warehouse at Fox Point, Providence, R. I. Branch offices of this firm are located in New York and Boston.

AMERICAN SALES AND PURCHASING CENTER FOR LEIPZIG FAIR

The establishment of a permanent American sales and purchasing center for American exporters and importers at the semi-annual Leipzig Sample Fair is being planned by the American Leipzig Fair Association, with headquarters in the Woolworth Building. Upward of 200,000 buyers from all countries of the world attended the last fair in March, while nearly 14,000 sellers, also from many lands, catered to them.

There were Americans among the more than 600 non-German manufacturers and jobbers who made use of the fair; but many of those lost all claim to special attention by operating through German agents and by scattering their booths about the grounds. An American center would gather them together and focus public attention upon them. At the American center, run by Americans in American fashion, the effort to get orders would be the first consideration. The fair people believe that an American national center would attract all regular visitors to the gathering, and in addition thousands of special visitors from many lands, who would be anxious to see our first important sales exhibition in Europe. Full information concerning the project may be obtained from the American Leipzig Fair Association, Woolworth Building, New York.

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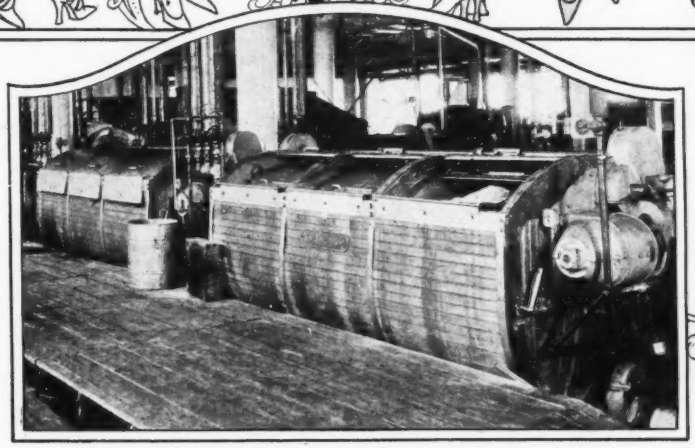

Battery of Smith-Drum rotary dyeing machines at Phoenix Hosiery Mills, Milwaukee, Wis. There are 34 Monel Metal machines in the Phoenix plant—all made and installed by SMITH-DRUM & CO. OF PHILADELPHIA, PA.

A SURVEY of the Phoenix dye house, made by the A. C. Nielsen Co., shows that the 18 Smith-Drum Monel Metal and Monel Metal lined dyeing machines in the Phoenix Milwaukee plant are doing the work of 36 wood machines and saving $24,510.20 a year in fixed costs, labor and floor space.

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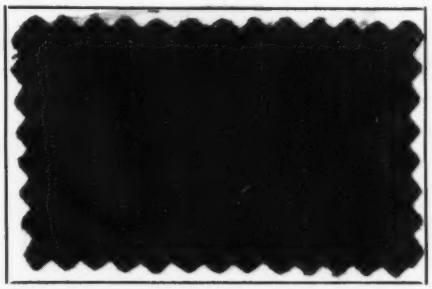
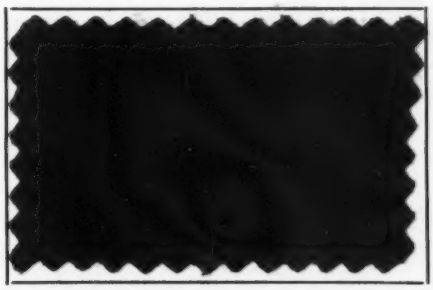
Akco Chrome Fast Green SW

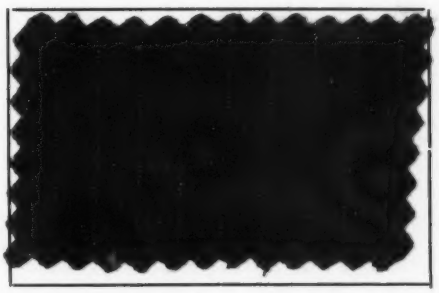


2½ lbs. Akco Chrome Fast Blue SW



5 lbs. Akco Chrome Fast Black SW

1 lb. 6 ozs. Akco Chrome Fast Blue SW
8 ozs. Akco Chrome Fast Yellow SW
6 ozs. Akco Chrome Fast Red SW
1 oz. Akco Chrome Fast Green SW



1 lb. 12 ozs. Akco Chrome Fast Yellow SW
14 ozs. Akco Chrome Fast Blue SW
10 ozs. Akco Chrome Fast Red SW
1 oz. Akco Chrome Fast Green SW

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AKCO Chrome Fast Brown
AKCO Chrome Fast Red SW
AKCO Chrome Fast Green GS
AKCO Chrome Fast Blue
AKCO Chrome Blue Black 6B
AKCO Chrome Fast Black EHK
AKCO Chrome Fast Black DM

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AKCOTHRENE Deep Green Ex. Paste
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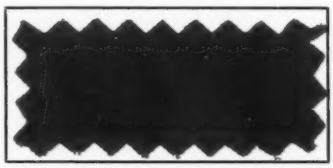
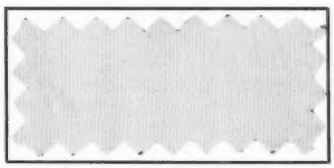


¾% National Niagara Sky Blue 6B

YELLOW

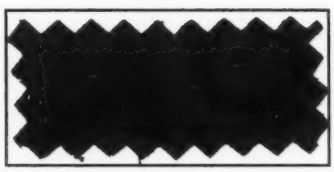


3% National Erie Yellow F

VIOLET

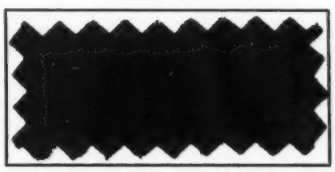
3% National Erie Violet 2 B with Developer B

RED



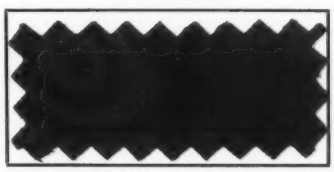
2½% National Erie Fast Red FD

BLACK



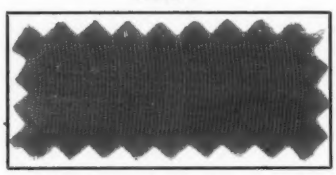
5% National Diazine Black DR with Developer DB

SCARLET



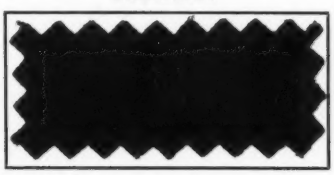
1½% National Erie Fast Scarlet 8 BA

GREY



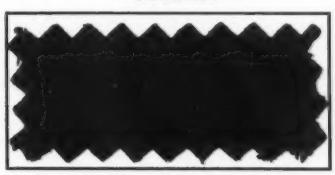
¼% National Erie Fast Grey M with Developer DB

BROWN



3% National Fast Brown 3 RB

GREEN



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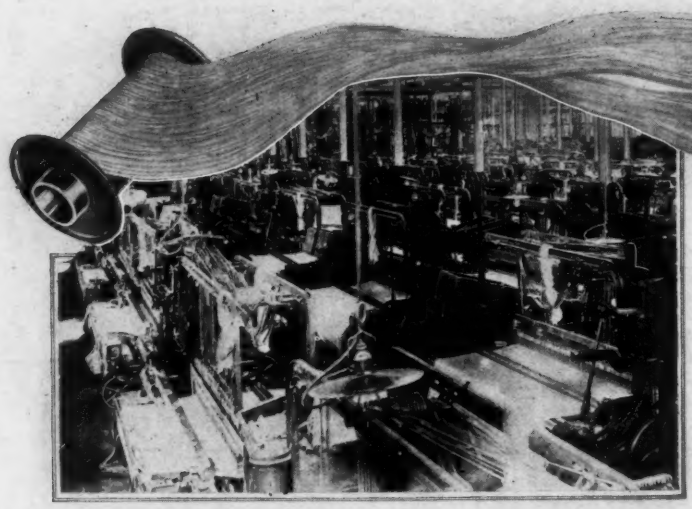

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